THE INFLUENCE OF GLASS TRANSITION TEMPERATURES ON THE PERFORMANCE OF ACRYLIC THERMOPLASTIC ADHESIVES

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SECTION I: INTRODUCTION

1.2 Introduction

Thermoplastic acrylic resins are widely used by conservators as adhesives, consolidants, and coatings due to their reversibility and stability. Extensive testing has confirmed that they are an ideal choice for use in conservation, as they are predicted to remain molecularly intact (and, therefore, stable and reversible) for hundreds of years.[1] Since stability and reversibility are two major tenets of the American Institute of Conservation for Historic and Artistic Works Code of Ethics as well as the codes of ethics of other conservation institutions around the world, the use of acrylic adhesives spread quickly among conservators.[2] The widespread and successful use of these adhesives inside museums has led many conservators to use them outdoors as well. However, while there has been extensive research on the use of acrylic adhesives in controlled environments, there are few scholarly articles that examine the effect of temperature — specifically, elevated outdoor temperatures — on

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[1] Robert L. Feller, Mary Curran, Catherine Bailie, "Photochemical Studies of Methacrylate Coatings for the Conservation of Museum Objects" In *Photodegradation and Photostabilization of Coatings* (Pittsburg: American Chemical Society, 1981) 194.

[2] The Code of Ethics & Guidelines for Practice of the American Institute for Conservation of Historic and Artistic Works, American Institute of Conservation (1994).; ICOM Code of Ethics for Museums, International Council of Museums (2004).

the long-term stability and performance of acrylic resins used as adhesives (i.e., rather than as a consolidant or a protective coating).[3]

One property that determines the flexibility or hardness of an acrylic resin upon setting is glass transition temperature (T_g); this is an important property to understand when using polymeric materials in outdoor environments. When the temperature of a thermoplastic acrylic resin is exceeded, the resin can eventually soften causing the adhesive to slump and flow.[4] Therefore, conservators need to consider an adhesive's T_g in order to make an informed decision about the appropriate adhesive for both their working environment and the long-term environmental conditions of a conserved object. This thesis presents the results of an experimental study that explores the use of acrylic resin adhesives within a particular set of environmental parameters and is relevant for conservators working on the restoration of historical objects and monuments in climates where the T_g of acrylic adhesives is regularly exceeded.

In order to understand the effect of elevated temperatures on acrylic resin adhesives, this thesis discusses the salient characteristics of each of the thermoplastic acrylic resins, along with the terracotta and limestone substrates used in the experimental portion of this study. The testing

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[3] N.B., conservators have used acrylic resins as consolidants for outdoor stone since the early 1960s. Poly(methyl methacrylate) (PMMA) and poly(buytl methacrylate) (PBMA) were used as both a consolidants and protective coatings on monuments in the mid-1960s, and by the end of the decade, Paraloid B-72 was being used extensively throughout western Europe as a consolidant for outdoor sculpture, monuments, and architecture. Cf. J.M. Cabrera Garrido, "The Portal of the Monastery of Santa Maria de Ripoll," *Monumentum* 1 (1967): 79-98.; P. Mora et al., "Consolidamento Provvisorio di un Marmo in Stato de Avanzata Degradazione," *Proceedings of the International Meeting, La Conservazione delle sculture all'aperto, Ente Bolognese Manifestazioni Artistiche, Bologna* (1969): 160-62.; Thomas C. Roby, "In Situ Assessment of Surface Consolidation and Protection Treatments of Marble Monuments in Rome of the 1980's, with Particular Reference to Two Treatments with Paraloid B-72," *Proceedings from the Eight International Congress on Deterioration and Conservation of Stone, Berlin, 30 Sept - 4 Oct. 1996* (Berlin: Möller Druck und Verlag,1996): 1015-1028.

The well-known "Bologna Cocktail," which is a mixture of Paraloid B-72 and a silicone resin, was developed in the 1970s as a consolidant for the marble sculptures of San Petronio Cathedral in Bologna and has since been studied extensively by conservation scientists as a treatment for outdoor stone. Cf. Gnudi, C. et al., Notizie sul restauro della facciata di S. Petronio (Bologna: Ministero per i beni culturali e ambientali 1979).; Marisa Laurenzi Tabasso, "Acrylic Polymers for the Conservation of Stone: Advantages and Drawbacks," *APT Bulletin Preservation of Historic Masonry* Vol 26 No 4 (1996): 17-21.; Wheeler, G.S., G.L. Shearer, S. Fleming, L.W. Kelts, A. Vega, and R.J. Koestler, "Toward a Better Understanding of B72 Acrylic Resin/Methyltrimethoxysilane Stone Consolidants," *MRS Proceedings* 185 (1990): 209-26.; Wheeler, G.S., Wolkow, E. and Gafney, H. "Microstructures of B72 Acrylic Resin/Mtmos Composites," *MRS Proceedings* (1992): 267.; M. Favaro et al., "Evaluation of Polymers for Conservation Treatments of Outdoor Exposed Stone Monuments. Part 1: Photo-Oxidative Weathering," *Polymer Degradation and Stability* 91 (2006): 3083-3096.

[4] Jane L. Down, "Solid Adhesive Properties" *Adhesive Compendium for Conservation,* Canada: Canadian Conservation Institute 2015), 21-22.

program included splitting terracotta and limestone core specimens in half before bonding them using one of six adhesives chosen for this study. The bonded cores and unbroken control groups were divided into two groups. Group A underwent temperature cycling, exposing the adhesives to a defined set of environmental conditions, followed by four-point bend flexural testing to determine the stiffness, bond strength, and the mode of failure — brittle vs. ductile — of each adhesive-bound core. The specimens in Group B were placed under a constant load and heat stressed in order to determine the thermally induced failure of each adhesive-bound core. The overall objective of this study is to understand the effect of elevated temperatures on acrylic adhesives in order to determine conditions under which these thermoplastic acrylic resins commonly used in conservation are appropriate for use as adhesives.

The goal of testing is to evaluate the strength and stability of these adhesives in response to high temperatures and temperature cycling while continuing to provide reversibility and adequate strength without increasing brittleness.

1.3 Literature Review

While many scientists and conservators have studied the properties of acrylic adhesives used as consolidants, coatings, and varnishes, little scientific testing has been done to evaluate T_g as it relates to strength and long-term stability of adhesives used for structural joints or fills.[5, 6] One significant contribution to the study of adhesives in conservation was the 1984 conference held by the International Institute for Conservation of Historic and Artistic Works entitled Adhesives and Consolidants. This conference, held in Paris over the course of six days, focused primarily on the standard practices of adhesives and consolidants used on various materials. However, only two case studies discussed the use of acrylic resins as adhesives: Carol Snow and Terry Drayman Weisser used Paraloid B-48N for structural repairs on Egyptian ivory sculptures and Thomas H. Taylor Jr. used Paraloid B-72 to repair the cracks in the architectural glass in situ at Colonial Williamsburg.[7]

In 1986, Stephen Koob published "The Use of Paraloid B-72 as an Adhesive: Its Application for Archaeological Ceramics and Other Materials" in which he proposes standardized method for preparing Paraloid B-72 as an adhesive. Koob pioneered the use of Paraloid B-72 as more than just a consolidant or surface coating, stating that its adhesive strength made it well-suited for use on a wide variety of materials.[8] According to Koob, the advantages of B-72 are its strength and hardness without brittleness and its higher T_g as compared to the polyvinyl acetate resins (B-72 has a T_g of

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5 The reconstruction of Tullio Lombardo's *Adam* included determining the glass transition temperature of the adhesives to ensure they were appropriate for use inside a museum environment.

6 Anon, "Acryloid Helps Preserve Art Treasures," *The Rohm & Haas Reporter* (1950): 14-15.; G. Olson and B Thordeman, "The Cleaning of Silver Objects," *Museum Journals* 50 (1951): 250-252.; Robert L. Feller, "Cross-linking of Methacrylate pol by Ultra-Violet Light," Papers at the New York Meeting, Division of Paint, Plastics, and Printing Ink Chemistry, American Chemical Society 17 No. 2 (1957).; Robert L. Feller, *On Picture Varnishes and their Solvents* (Cleveland: Press of Case Western Reserve University, 1959). Robert L. Feller, Mary Curran, Catherine Bailie, "Photochemical Studies of Methacrylate Coatings for the Conservation of Museum Objects," In *Photodegredation and Photostabilization of Coatings* (Pittsburg: American Chemical Society, 1981), 183-195.; A.E. Werner, "Plastics Aid in Conservation of Old Painting," *British Plastics* 25 (1952): 363-366.; A. Zappalà and P. La Mendola, "A Method of Preparing and Using an Acrylic Resin Coated Paper," In *5th Triennial Meeting: Zagreb, 1-8 October 1978: Preprints*, International Council of Museums Committee for Conservation (1978).; E.C. Welsh " A Consolidant Treatment for Powdery Matte Paint," In *Annual Meeting: Proceedings, American Institute for Conservation* 8 (1980): 141-150.; M. Serek-Dewaide, "Disinfection and Consolidation of Polychromed Wood at the Institute Royal du Patrimonine Artistique, Brussels," In *Conservation of Wood in Painting and the Decorative Arts, Oxford Congress, 17-23 September, 1978: Preprints* (International Institute for Conservation 1978), 81-83.; V. Von Reventlow, "Use of B-72 in the Restoration of a Marquetry Surface: Case History," In *Conservation of Wood in Painting and the Decorative Arts, Oxford Congress, 17-23 September, 1978: Preprints* (International Institute for Conservation 1978), 37-39.

7 Carol E. Snow and Terry Drayman Weisser, "The Examination and Treatment of Ivory and Related Materials," In *Adhesives and Consolidants: preprints of the contributions to the Paris Congress, 2-8 September 1984* (London: IIC, 1984), 141 -145.; Thomas H. Taylor Jr. "In Situ Repair of Architectural Glass," In *Adhesives and consolidants: preprints of the contributions to the Paris Congress, 2-8 September 1984* (London: IIC, 1984), 202-204.

8 S.P. Koob,"The Use of Paraloid B72 as an Adhesive: its Application for Archaeological Ceramics and Other Materials," *Studies in Conservation* 31 (1986): 7.

40º C, PVA AYAT 28º C). In addition to a detailed explanation for preparing B-72, Koob discussed the importance of proper application with particular emphasis on ceramic materials.[9] This publication is still considered a standard in the conservation field when it comes to the preparation and use of Paraloid B-72 as an adhesive.

One year later, in 1987, Velson Horie published *Materials for Conservation: Organic Consolidants, Adhesives, and Coatings*, which was updated in 2010. This book is an encyclopedic guide to polymer science and various adhesives that are commonly used in conservation. In the early chapters of the book, Horie discusses how the polymer's structure affects the adhesives' physical and mechanical properties. In regards to T_g, Horie explains that the mechanical property of stiffness (modulus of elasticity) changes dramatically around the T_g of thermoplastic adhesives — below the T_g the adhesive is solid but when the temperature rises above the T_g the adhesive will succumb to cold flow. The temperature at which the adhesive turns to a soft, elastic substance and begins to flow is determined by the molecular weight and interactions of the polymer chains that make up the acrylic resin.[10]

In 2001, Jerry Podany et al. conducted two studies on the use of acrylic resin adhesives for reassembly of large stone sculpture. In their article "Paraloid B-72 as a Structural Adhesive and as a Barrier within Structural Adhesive Bonds: Evaluations of Strength and Reversibility," the authors discuss the historical use of epoxy resins as adhesives in sculpture conservation, along with the major drawbacks associated with thermosetting epoxy resins in terms of reversibility. Their study evaluates the use of B-72 as an adhesive and as an interface barrier, along with the more commonly used structural adhesives, epoxy and polyester, from the standpoint of shear and tensile strength, as well as reversibility. Two important findings came from this study: first, researchers determined that B-72 used as a barrier layer for polyester or epoxy resin adhesives is sufficiently strong enough for

[9] ibid 13.

[10] Velson Horie, *Materials for Conservation: Organic Consolidants, Adhesives and Coatings*, Routledge, London (2010) 17-22.

structural joints. By using a barrier layer of B-72 between the marble and the epoxy, the epoxy resins were made reversible without any decrease in strength to the joint. The second finding was that in some solutions, B-72 alone is strong enough under tensile load for use as a structural adhesive, thus maintaining the reversibility of the joint.[11]

In 2006, M. Favaro et al. published a two-part paper on the "Evaluation of Polymers for Conservation Treatments of Outdoor Exposed Stone Monuments. Part 1: Photo-Oxidative Weathering." Favaro et al. focused on the use of Paraloid B-72, Paraloid B-67, and a silicone-based product — Dri-Film 104 — as consolidants for stone monuments. This paper produced important research on the deterioration process of acrylic and silicone-based resins in simulated outdoor environments. However, the study focuses on the use of these resins as consolidants and not as adhesives. In this study, the research team used two types of weathered marble to test the consolidation properties of the resins. Their findings showed that all of the polymer resins underwent structural changes as a result of the artificial weathering by photo-oxidation. These structural changes included partial cross-linking in the silicone polymers caused by hydrolytic processes, chain scissions, and/or reticulated structures in the acrylic resins due to photo-oxidation. Modifications to the polymer structure, such as cross-linking, limit the reversibility and removability of the polymer from treated surfaces.[12] In future research, the team plans to replicate the environments of specific locations, such as Venice, to see how these polymers react before they are used on outdoor marble sculptures.[13]

At Columbia University, Mersedeh Jorjani examined the use of adhesives for marble repair in her master's thesis in 2007. In her research, Jorjani looked at interfacial fracture and bond line width

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[11] Jerry Podany, Kathleen M. Garland, William R. Freeman and Joe Rogers, "Paraloid B-72 as a Structural Adhesive and as a Barrier within Structural Adhesive Bonds: Evaluations of Strength and Reversibility," *Journal of the American Institute for Conservation*, Vol 40 No 1 (Spring, 2001): 15-33.

[12] Favaro, M., R. Mendichi, F. Ossola, U. Russo, S. Simon, P. Tomasin, and P.A. Vigato. "Evaluations of Polymers for Conservation Treatments of Outdoor Exposed Stone Monuments. Part I: Photo-Oxidative Weathering," *Polymer Degradation and Stability* 91 (2006): 3083–96.

[13] ibid 3095.

in both thermosetting and thermoplastic adhesives. Using tensile splitting, she tested samples of Carrara marble, which had been glued together in the so-called Brazil disk sandwich, in order to determine the interfacial fracture toughness. Her results showed a surprising similarity between the performance of the thermosetting and thermoplastic adhesives, all of which were found to be strong enough for use on freshly fractured Carrara marble.[14] The second phase compared bond widths of adhesives in smooth and fractured surfaces with previous experiments conducted by Susan Bradley in 1984 and Jerry Podany et al. in 2001.[15] The largest bond width observed was with the fractured B-72/Epotek 301-2 sandwich. Epotek 301-2, which is a low viscosity epoxy resin adhesive, is often used with B-72 to create reversible structural joints.[16]

The interfacial fracture toughness results first described by Jorjani in 2007 were published in greater detail as Jorjani et al. 2009 and as Rahbar et al. in 2010. In the 2009 article, the analysis compared toughness of thermoplastic and thermosetting adhesives used with Carrara marbles samples. Researchers found that the fracture toughness increased with increasing mode mixity, except for polyvinyl butyl adhesives.[17] Rahbar et al. examined mode mixity, also called the phase angle of fracture, which is the relative proportion of tractions ahead of the crack tip in sliding mode (mode II) and opening mode (mode I) in the fracture. The increase of fracture toughness with mode mixity generally observed is attributed mainly to the presence of surface forces that inhibit the sliding of crack surfaces.[18] The results also showed that the interfacial toughness values were higher for the

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[14] Mersedeh Jorjani, "An Evaluation of Adhesives Used For Marble Repair," Columbia University (2007): 26.

[15] Susan Bradly, "Strength testing of adhesives and consolidants for conservation purposes," *Studies in Conservation* Vol 29 Issue 1 (1984): 22.

[16] Jorjani 32.

[17] Jorjani, Mersedeh et al., "An Evaluation of Potential Adhesives for Marble Repair." In *Holding it All Together: Ancient and Modern Approaches to Joining, Repair, and Consolidation*, London: Archetype in association with the British Museum (2009) 143–49.

[18] Rahbar et al., "Mixed Mode Fracture of Marble/Adhesive Interfaces," *Material Science and Engineering:* A 527 (2010): 5.

pre-fractured samples compared to the smooth samples for all three types of adhesives — thermosetting, thermoplastic, a combination of thermosetting and thermoplastic. [19]

In 2011, Ting Tan et al. published an article as part of the restoration of Tullio Lombardo's *Adam* at the Metropolitan Museum of Art on the interfacial creep crack growth behavior along the interfaces of different adhesives and marble. Following up on research begun by Andrea Buono in 2009 for her master's thesis at Columbia University, the research team examined the possibility for sub-critical crack growth to occur in Carrara marble, at the interfaces between adhesives and the marble, under static loading conditions.[20] Since resins can creep at room-temperature, the team was concerned that the creep crack growth would cause sub-critical fracturing.[21] Their results showed that the adhesives that were a combination of thermosetting and thermoplastic resins had slower crack growth rates, while the crack microstructure interactions, specifically in the B-72/B-48N specimens, showed evidence of crack initiation along the interface between the Carrara marble and the adhesive.[22] Researchers also ran models to predict the service life of the adhesives. The predictions suggested the lives of the B-48N fractured specimen and the B-72 smooth specimen would last several thousand years, which is consistent with the life spans of several large-scale marble sculptures. However, the predicted lives obtained for Paraloid B-72:B-48N fractured and smooth samples were over 10,000 years. Ultimately, the B-72/Epotek sandwich samples emerged as the system with the best combination of slow crack growth rate and predicted structural life, suggesting that a combination of thermoplastic and thermosetting adhesives may provide the best option for marble structure restoration.[23]

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[19] ibid 7.

[20] Andrea Buono, "Adhesives for Marble and Their Creep Behavior," Columbia University (2009).

[21] Ting Tan et al., "Sub-Critical Crack Growth in Adhesive/Marble Interfaces," *Materials Science and Engineering*: A 528 No. 10–11 (2011): 3697.

[22] ibid 3701.

[23] ibid 3701.

In 2014, the conservation team at The Metropolitan Museum of Art published their extensive research and findings on various adhesives tested for the reconstruction of Tullio Lombardo's *Adam*.[24] The project involved the reconstruction of a life-size Carrara marble sculpture that broke into twenty-eight large pieces and hundreds of smaller fragments after its supporting pedestal collapsed. The goals of the adhesive testing were: (1) to evaluate the adhesive's strength and stability; (2) to determine the degree of displacement caused by the adhesive system; (3) to assess reversibility. As discussed, epoxy resins have traditionally been used for large-scale sculpture reconstruction because of their strength; however, the problem with thermosetting epoxy resins is that they are not reversible, they have excess strength compared to the marble and, if used with a reversible, thermoplastic barrier layer (such as B-72), the thickness of the joint may cause displacement. The team was therefore interested in using thermoplastic acrylic resins, specifically B-72 and B-48N, for the chemical stability, reversibility, and thin bond line. In collaboration with Columbia and Princeton Universities, the museum's team tested the interfacial fracture toughness in order to assess the strength of nine adhesive systems. The adhesive that performed best overall was a 3:1 mixture of B-72:B-48N. Additionally, their findings indicated that in general, thermoplastic acrylic adhesives are nearly as strong as the thermosetting epoxy resin adhesives, and that all of the systems have high enough strength for use with Carrara marble.[25]

Another important factor in choosing an adhesive for the reconstruction of *Adam* was the thickness of the bond line. The high quality of the Carrara marble and the cleanness of the breaks meant that the fragments would fit together tightly. Therefore, the bond line (i.e., the space occupied by adhesives at each join) had the potential to cause displacement, preventing proper reconstruction. For example, the right leg had broken into three pieces and the left leg into five. As

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24 N.B., the Jorjani 2007, Jorjani et al. 2009, Buono 2009, Rehbar et al. 2010, and Ting Tan et al. 2011 were part of the research conducted for the Tullio reconstruction project.

25 Carolyn Riccardelli, Michael Morris, George Wheeler, Jack Soultanian, Lawrence Becker, and Ronald Street. "The Treatment of Tullio Lombardo's Adam: A New Approach to the Conservation of Monumental Marble Sculpture," *Metropolitan Museum Journal* 49 No. 1 (2014): 68.

the conservators bonded the fragments back together, the displacement from the joins in each leg would be additive and could therefore cause misalignment at the final connection point due to the unequal number of joins.[26] Fortunately, the results of the B-72:B-48N mixture produced a bond line thickness lower than the epoxy resin and barrier layer, and was within the acceptable thickness range.[27]

Lastly, the team examined the long-term stability of the adhesives with regards to creep. This research, in collaboration with Columbia and Princeton Universities, was the first scientific study of creep conducted on conservation materials. As discussed above in Ting Tan et al., the study found that thermoplastic acrylic resins (B-72, B-48N, and the B-72:B-48N blend) performed as well as the thermosetting epoxy resins. Additionally, the service life of the B-72:B-48N blend is several thousands of years and analysis of the results suggests that by adding B-48N to a B-72 adhesive, it may prevent long-term creep.[28]

In 2015, the Canadian Conservation Institute published Adhesive Compendium for Conservation, which is the most extensive examination of adhesives and the mechanical and structural properties since Horie's book in 2010. In this book, Jane L. Down et al. examine the physical and chemical properties of various natural and synthetic polymers used in adhesives. Chapter four is especially relevant, as Down discusses T_g and how it can be manipulated and lowered with the addition of plasticizers, solvents, water, or adhesives with a lower T_g value. Chapter six is an extensive list of adhesives including their chemical makeup, setting mechanisms, T_g, preparation methods, history, and use in conservation.[29] The second half of the book explores the use of adhesives in the conservation of various materials. Stephen Koob's chapter on ceramics and glass advocates the use of Paraloid B-72 over epoxy resins due to its stability, reversibility, and fast setting

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[26] ibid 67.

[27] ibid 69.

[28] ibid 69.

[29] Down 21-25, 35-116.

time.[30] While Koob does not directly discuss the effects of temperature on long term stability of the adhesives, he does mention that consolidation and adhesion should be carried out in a climate controlled environment, preferably around 18-24 ^{o}C with 35-55% relative humidity. Koob also mentions that higher temperatures will result in rapid evaporation of the solvent, which will cause poor consolidant penetration or improper film formation, while extremely low temperatures will slow down the evaporation of the solvent and allow the consolidant to essentially freeze.[31]

The final section in Down's book examines the use of adhesives in the conservation of stone. In this section, George Wheeler discusses how epoxy resin adhesives were adopted by the conservation field early on due to their strength. When working with dense materials, such as stone, strength of adhesion is of great importance. However, Wheeler recognizes that epoxy resin adhesives do not allow for reversibility, which is a critical problem for conservators. Like Koob, Wheeler recommends the use of acrylic resins as structural adhesives that can be used in combination with traditional dowels and pins. In addition to discussing the strength of acrylic adhesives, Wheeler addresses the important issue of environmental considerations when deciding on an appropriate adhesive. He states that the most important considerations when picking an adhesive for outdoor use are hydrolytic stability and T_g.[32]

1.3 Brief History of Adhesives Used in this Study

See Appendix 1 for a more extensive history of Rohm and Haas

The majority of acrylic resins are synthesized from acrylates derived from acrylic acid, and methacrylate derived from methacrylic acid. These two acrylic monomer units copolymerize with an

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[30] Stephen Koob, "Ceramics and Glass" In *Adhesive Compendium for Conservation* (Canada: Canadian Conservation Institute, 2015), 191.

[31] ibid 190.

[32] George Wheeler, "Stone" In *Adhesive Compendium for Conservation* (Canada: Canadian Conservation Institute, 2015), 199 - 202.

alkyl ester group — methyl, ethyl, butyl, etc. — to form the polymers that make up the Paraloid series of resins.[33]

A. Paraloid B-72 — an ethyl methacrylate and methyl acrylate copolymer

In 1941, The Resinous Products & Chemical Co. published a booklet on all of their synthetic resins. B-7, B-72, and B-75 are recommended as a consolidant, clear finish for metal, and white enamel respectively. B-72 was supplied as a 40% solution in tuluol only and the softening temperature was reported as 35-55°C.[34] It was developed as a resin solution particularly suited for use in pigmented finishes, but the suggested uses included: 1) clear coatings on metal for resistance to fruit acids, moisture, etc.; 2) clear coatings on metal to prevent discoloration or tarnishing — it is suggested for use on silverware, trophies, and decorative polished metal; 3) pigmented coatings, where good resistance to alkali or acids, as well as good initial color and color retention, is desired; 4) protective coating for metal trimming on automobiles, copper screens, chromium, stainless steel store fronts, etc. [35]

In 1942, Acryloid B-73 was developed for the military as an acrylic resin binder for luminescent paints. The Acryloid resins were successful as a vehicle for pigments because they dried rapidly, were not affected by rain, held their luminosity for several hours, and remained stable after several months in storage.[36] In 1943, Resinous Products claimed that the Acryloids, which included B-73 and B-72, were "especially suitable for luminescent pigments because of their water-white color, non-yellowing, non-oxidizing, non-reactivity qualities and their resistance to water."[37] They

[33] Down 81.

[34] Synthetic resins: Amberol, Duraplex, Amberlac, Paraplex, Uformite, Acryloid, Aquaplex, Oilsolate; plywood adhesives, special products (Philadelphia: Resinous Products & Chemical Co, 1941), 53.57.

[35] ibid 58.

[36] "Acryloid B-73 for Luminescent Paints," *The Resinous Reporter* Vol III, No. 1 (1942).

[37] "Acryloids for Luminescent Paints," *The Resinous Reporter* Vol IV No. 3 (1943).

recommended that the paint film should contain 50-60% luminescent pigment and the remainder should be one of the two Acryloids; they suggested applying two coats of the paint and then a clear coat of one of the Acryloids over top. For the top coat, the Acryloid should be used at a 20% dilution for brushing or 15% for spraying.[38]

However, following WWII, Resinous Products needed a new market for their Acryloid resins. Though they continued to be promoted as a binder for luminescent paints, in 1947, two issues of *The Resinous Reporter* highlighted the use of Acryloids in art restoration. The first article was brief, but boldly claimed that the Acryloids had recently been used "successfully" as a "stain-proof coating for building stone," and that Acryloid B-72 specifically could be used to form a thin, clear protective coating on silver, perishable drawings, and lampshades.[39] Though the most significant endorsement of B-72 as a tool for art conservators was in an article entitled "China-Repair Employs Synthetic Resins: Acryloid B-72 and Uformite 500 Used by Philadelphia Artist in Restoring Objet d'Art." A. Ludwig Klein & Sons was a private art restoration company specializing in the repair and restoration of porcelain. In the article, Mr. Klein explained that Acryloid B-72 provided a high-gloss coating necessary to achieving a match between his porcelain paste fills and the original porcelain finish. Using Acryloid B-72 as a protective coating on unglazed porcelain reduced the "dust-catching-ability" and allowed for coated objects to be dusted and washed without damage to the surface or gloss. In his experiments, Mr. Klein found that B-72 worked well as a waterproof protection varnish for highly glazed white porcelain, wood, metal, and leather. The most important part of this article is that Mr. Klein was selling prepared kits, complete with instructions for use, which contained "wax and plaster, porcelain paste, brushes and pigments, and small quantities of the synthetic resins, Uformite 500 and Acryloid B-72" to antique dealers, china shops, and museums.[40] This brief article,

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[38] ibid.

[39] "Acryloids Available: Expanded Production Meets Enlarged Demand for Acrylic Ester Resins," *The Resinous Reporter* Vol VIII No. 3 (1947): 6-7.

[40] "China-Repair Employs Synthetic Resins: Acryloid B-72 and Uformite 500 Used by Philadelphia Artist in Restoring Objet d'Art," *The Resinous Reporter* Vol VIII, No. 5 (1947): 8-9.

in a chemical company's monthly publication, is an important insight into how Paraloid B-72 became the most prevalent coating/consolidant/adhesive used in conservation.

Today, Paraloid B-72 is regarded as a resin suited for almost every sub-discipline in conservation. It is considered a Feller Class A material (Table 1) — meaning it is of "excellent" quality, suitable for conservation use, and should last at least 100 years — and is used by conservators as a coating, consolidant, and adhesive.[41] The suggested uses for Paraloid B-72 include hot melt adhesive for paper, consolidant for matte pigments, consolidant for lacquer work and wood, facing for marquetry, and coatings for iron.[42] Despite our ability to design polymers with more appropriate properties for conservation, the popularity and universal use of Paraloid B-72 has spread among conservators as it remains one of the few polymers tested and cleared for conservation use.

Table 1: Feller's Standards of Intended Use and Photochemical Stability for Materials in Conservation[43]

Class	Classification	Intended Useful Lifetime	Approximate Equivalent Standard of Photochemical Stability
T	Materials in temporary contact	Less than 6 months?	-
C	Unstable or fugitive	Less then 20 years	BS1006 class 3 or less
B	Intermediate	20-100 years	3 to 6
A	Excellent	(A2?) greater than 100 years	Greater than BS1006
		(A1?) greater than 500 years	?

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41 R. L. Feller "Thermoplastic Polymers Currently in Use as Protective Coatings and Potential Directions for further Research," *AICCM Bulletin* 2 Vol 10 (1984): 5-18.

42 A. Zapapala and P. La Mendola, "A Method of Preparing and Using an Acrylic Resin Coated Paper," In *5th Triennial Meeting: Zagreb, 1-8 October 1978: Preprints* (International Council of Museums Committee for Conservation, 1978).; E.C. Welsh, "A Consolidant Treatment for Powdery Matte Paint," In *Annual Meeting: Proceedings* (American Institute for Conservation, 1980).; M. Sawada, "Zur Konservierung eines bemaltenjapanischen Lackgefaesses," *Arbeitsblätter für Restauratoren* 14 (1981).; M. Serck-Dewaide, "Disinfection and Consolidation of Polychromed Wood at the Institute Royal du Patrimonine Artistique, Brussels" In *Conservation of Wood in Painting and the Decorative Arts, Oxford Congress, 17-23 September, 1978: Preprints* (International Institute for Conservation, 1978).; V. von Reventlow, "Use of B-72 in the Restoration of a Marquetry Surface: Case History," In *Conservation of Wood in Painting and the Decorative Arts, Oxford Congress, 17-23 September, 1978: Preprints* (International Institute for Conservation, 1978).; G. Evers, "Restoration and Reconstruction Problems Taking as an Example the Helmet from Niederralta," *Arbeitsblätter für Restauratoren* 1 (1968).

43 R.L. Feller, *Accelerated Aging Photochemical and Thermal Aspects* (Marina del Rey, CA : Getty Conservation Institute, 1994), 7.

B. Paraloid B-48N — a methyl methacrylate and butyl acrylate copolymer

Paraloid B-48N is also used primarily as a clear or pigmented coating for bare metals. It is known to have good adhesion, durability, toughness, flexibility, and weathering characteristics, but has also been reported to have issues of yellowing and discoloration.[44, 45] However, in 1981, Feller found that acrylics containing butyl, such as Paraloid B-48N, as well as amyl esters have a greater tendency to cross-link and that the likelihood of cross-linking increases as the temperature increases.[46]

Conservation treatments with acrylic resins have been reported for wood, paper, mosaics, pigments, lacquerware, amber, fossils, ceramics, glass, and stones. Such uses specifically site the strong adhesion and water-repellent properties of acrylic resins, yet at the same time they require environmental stability, especially when the resins are intended to act as consolidants or protective coatings on monuments and works of art exposed to outdoor conditions. [47]

C. Paraloid B-44 — a methyl methacrylate and ethyl acrylate copolymer

Paraloid B-44 is used most often as a protective coating for metals, and as such it forms the basis of INCRA's (International Copper Research Org.) Incralac. Incralac is used by conservators as a transparent coating for copper and silver alloy objects, especially on outdoor sculpture, because of its resistance to weathering and the incorporation of a corrosion inhibitor, benzotriazole.[48] While Paraloid B-44 has a higher glass transition temperature than Paraloid B-72, Erhardt et al. found that

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[44] Chandra L. Reddy et al, "Evaluation of three protective coatings for indoor silver objects," *Objects Specialty Group Postprints* Volume 6 (1999): 41-44.

[45] In 1992, the Canadian Conservation Institute found that after naturally aging both B-48N and B-72 for five years in light and dark environments for a period of three to five years, that the B-48N exhibited less yellowing than the B-72. (Down et al 1992)

[46] Feller (1981) 194.

[47] Chiantore et al (2000) 17.

[48] Horie 164.; A. Moncrieff, "Protecting Silver from Tarnishing," *In International Institute for Conservation of Historic and Artistic Works Newsletter* 4 No 2 (1966): 6-7.

after ten years of outdoor exposure on sculpture, Incralac becomes insoluble presumably due to cross-linking.[49]

D. Paraloid A-11 — methyl methacrylate polymer

In 1941, The Resinous Products & Chemical Co. published a booklet on all of their synthetic resins. A-10 is listed as a clear finish and heat-resistant enamel recommended for use where maximum resistance to alcohol, coal tar, hydrocarbons and moisture is needed. The suggested uses specifically mention that A-10 is the hardest of all the Acryloids and can be used as a heat-resistant film and enamel. As a clear coating, A-10 gives an excellent resistance to petroleum and coal-tar hydrocarbons, including coating for paper cap linings and paper boxes.[50]

Three years later, in 1944, *The Resinous Reporter* printed an article introducing phosphorescent paints made with a new product called Acryloid A-10 (presumably the predecessor to A-11). These paints were developed by The Aunt & Wiborg Division Interchemical Corporation in Cincinnati, Ohio as a paint that could "withstand the rough handling and severe weather conditions of the many battlefronts."[51] "When a sharpshooter draws a bead on the enemy in the steaming jungles of the Pacific or in the mountains of Italy, his aim will be improved by a tiny spot of glowing light on his rifle sight."[52] These quotes are interesting because they both focus on the stability of the paints in extreme environments. The Aunt & Wiborg Division Interchemical Corporation chose A-10 because of its "excellent stability with phosphorescent pigments, clarity,

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[49] David Erhardt, Walter Hopwood, Tim Padfield and Nicholas Veloz, "The Durability of Incralac: Examination of a ten-year old treatment," In *7th Triennial Meeting: Copenhagen, 10-14 September 1984: Preprints* (Paris: International Council of Museums in association with the J. Paul Getty Trust, 1984): 1-3.

[50] *Synthetic Resins* 57.

[51] "Phosphorescent Paint Improves Marksmanship of Snipers: Ault and Wiborg Division of Interchemical Corp. Develop New Finish with Acryloid," *The Resinous Reporter* Vol V, No. 1 (1944) 9.

[52] ibid 9.

speed of dry, adhesion to metals, film hardness, good oil resistance, non-yellowing characteristics, and general durability."[53]

In the 1947 *The Resinous Reporter*, Acryloid A-10 continued to be recognized as the hardest of the Acryloid group and was therefore advertised as an appropriate choice for heat-resistant white enamels. Specifically, *The Resinous Reporter* claimed that A-10 could be sprayed over a heat-resistant primer, such as Duraplex ND-77B and then baked for 20 minutes at 400°F. A-10's resistance to heat, scouring, soap, and grease along with its "permanent gloss and adhesion" made it an ideal for stoves (both gas and kerosene) and electric roasters.[54]

Today Paraloid A-11 is still considered one of the harder resins in the Paraloid series, though it has since been surpassed by A-21.[55] Paraloid A-11 is suggested for use as a coating for metal, vinyl, and plastic.[56] There are no published case studies or experiments concerning Paraloid or Acryloid A-11 in the conservation literature. The one exception is the Caroline K. Keck Collection at The Winerthur Library. Within the archive, Folder 16 in Box 16 is entitled "Microcrystalline Waxes and Acryloid A-11." It would be interesting to investigate this further, considering Mrs. Keck's "wax-resin" treatment for paintings has been discussed widely among conservators.[57]

E. Paraloid B-72:B-48N in a 3:1 mixture

Objects conservator Donna Strahan began an empirical study of an adhesive mixture using Paraloid B-72 and B-48N during an excavation at Troy in the late 1990s while working on the reconstruction of large ceramic storage jars known as pithoi. The jars are approximately five feet tall

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[53] ibid 9.

[54] "Acryloids Available," 6-7.

[55] Ultimate Hardness of Clear Films (KHN) for Paraloid A-21 = 21-22
Ultimate Hardness of Clear Films (KHN) for Paraloid A-11 = 18-19

[56] "Paraloid A-11," The Dow Chemical Company, http://www.dow.com/en-us/markets-and-solutions/products/PARALOIDA/PARALOIDA11.

[57] Caroline K. Keck, "Lining Adhesives: Their History, Uses, and Abuses," *Journal of the American Institute for Conservation* Vol 17 No. 1 (1977): 45-52.

and the walls are just over an inch thick, making them very heavy. The adhesive choice was of paramount importance because a few of the pithoi had been selected for an exhibition in Germany from 2001-2002. This meant the jars needed a high degree of reconstruction that would last through transport, be of high enough quality for exhibition, and then able to withstand outdoor storage in Turkey with only a metal roof for protection. Ultimately, because of the excessive weight and extreme climate fluctuations (temperatures can range from below 0 - 30° C), Strahan conducted an empirical experiment with a new adhesive mixture using B-72 and B-48N as a way to manipulate the T_g. This mixture provided the necessary strength and stiffness for the high temperatures while maintaining the possibility for future reversibility. In 2008, the pithoi were re-examined and found to still be in good condition.[58] This mixture was subsequently tested in the Tullio project and was chosen as the primary adhesive for the *Adam* sculpture.[59]

1.4 Adhesive Properties

A. Thermoplastic Polymers

In the field of conservation, several properties of adhesives must be considered before use. This research focuses on the important property of glass transition temperature for thermoplastic polymeric materials. Thermoplastic polymers, as compared to thermosetting polymers, are made up of amorphous long-chain polymers, which are held together by relatively weak secondary bonds between neighboring molecules. Thermosetting polymers, when fully cured, consists of a continuous network in which molecules are held in place by covalent bonds. Because the molecular chains in thermosetting polymers are not chemically bonded to one another, the molecules are able to easily slide over each other when the temperature increases or when they are dissolved in a solvent. As a result, when a thermoplastic material is heated, it becomes soft and will flow. Once the

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58 Donna Strahan and Simone Korolnik, "Archeological Conservation," *Studia Troica Monographien* 5 (2014): 521-523.

59 Riccardelli et al. 70.

thermoplastic material is allowed to cool it becomes rigid but, if reheated, it will once again soften.[60] For a conservator, using a thermoplastic adhesive means that if the adhesive becomes too soft, it can begin to show deformation, resulting in sagging or eventual failure of the bond in an object. Conversely, if the adhesive is too hard and brittle it can crack or fail in response to movement in an object.

B. Glass Transition Temperature

The temperature at which the thermoplastic material changes from a rigid solid to a soft, pliable state is called the glass transition temperature (T_g). T_g is a second-order transition, meaning that unlike melting, which is a first-order transition, the heat capacity continues to increase with the rising temperature but does not involve a latent heat. As seen in Figure 2, the T_g is the primary transition for amorphous polymers and melting and boiling are the primary first-order transitions for crystalline polymers. The T_g of polymers varies widely and is affected by the molecular weight (or chain length), composition and structure of the polymer, hydrogen bonding, secondary bonding, the presence of chemical chain defects such as cross-linking and scission, the presence of the side groups, and plasticizers.[61]

A general rule in understanding the effects of chemical and physical variables on an adhesive's T_g, any structure that reduces chain mobility will increase the T_g. Therefore, the T_g of a polymer is directly related to its molecular weight and backbone flexibility. Simply put, the higher the molecular weight, the higher the T_g. This is because larger molecular chains have less freedom of movement and, therefore, require more thermal energy to allow for movement between the molecules. Additionally, the more flexible the backbone chain is, the better the polymer will move, and the lower its T_g will be.

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[60] Science for Conservators, Volume 3: Adhesives and Coating (London and New York: Museums & Galleries Commission and Rutledge, 1992), 38-39.; Down, 3.

[61] Michael R. Schilling, "The Glass Transition of Materials Used in Conservation," *Studies in Conservation* Vol 34 No. 3 (1989): 110.

Figure 1: Glass Transition Temperature[62]

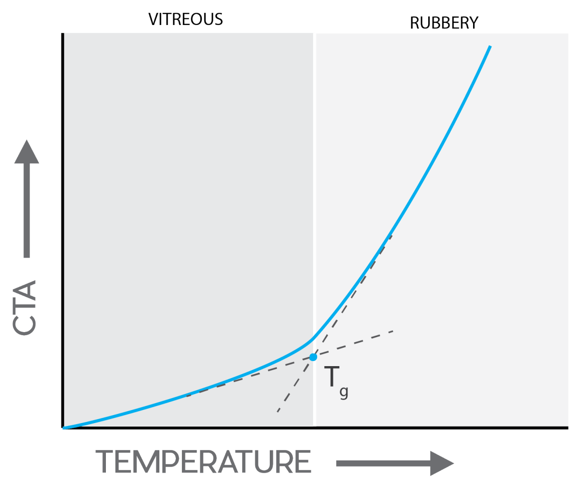
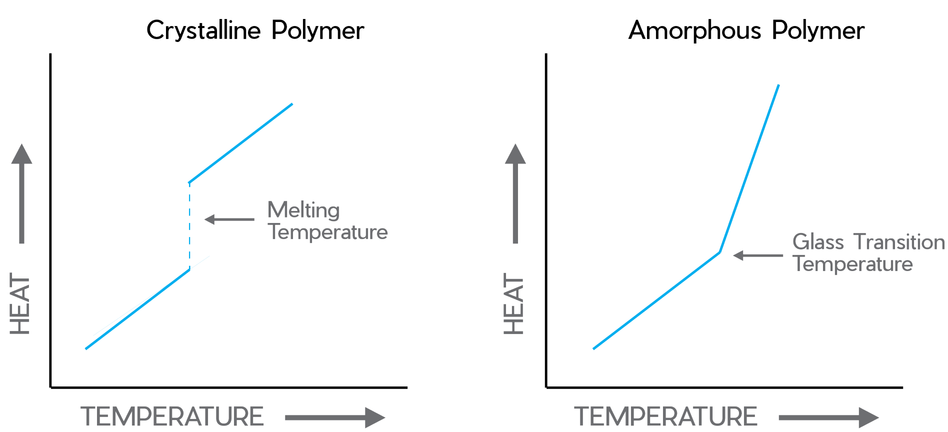


Figure 2: Heat vs. Temperature for a Crystalline and Amorphous Polymer[63]

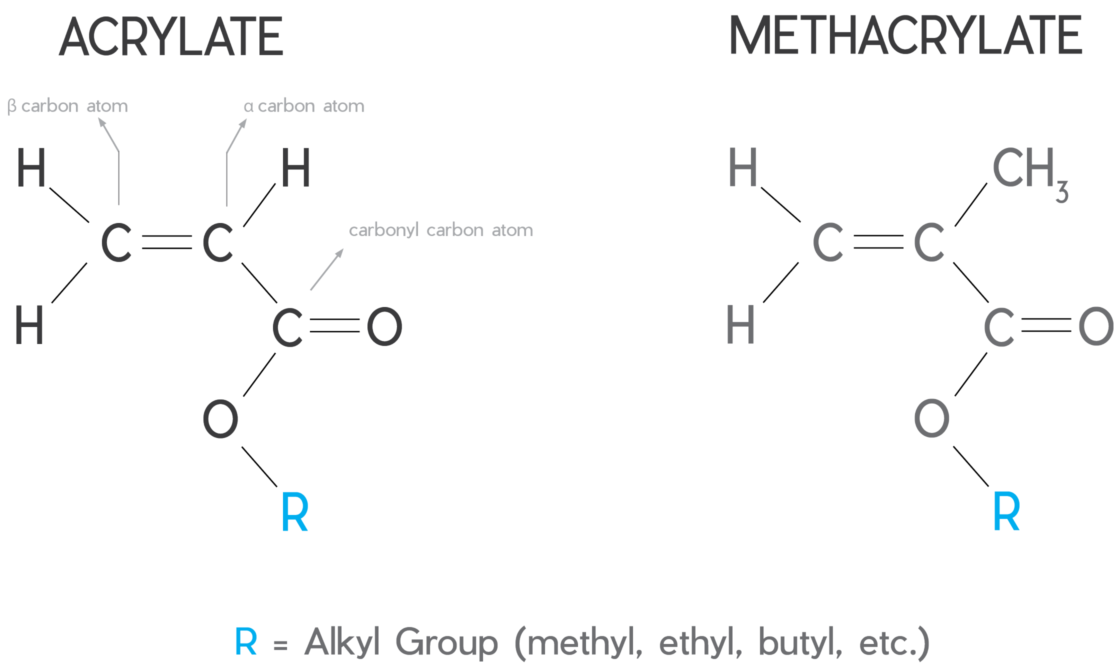


62 "How To: Optimizing Glass Transition Temperature (Tg)," Master Bond Inc. http://www.masterbond.com/techtips/how-optimizing-glass-transition-temperature-tg.

63 "The Glass Transition," *The Polymer Science Learning Center*, http://pslc.ws/macrog/tg.htm.

The composition, length, and flexibility of the side chains also play a role in determining the overall chain flexibility of the polymer. Acrylate polymers have a hydrogen atom adjacent to the alpha carbon from the carbonyl group (Figure 3). This allows for more rotational freedom than the methacrylate, as it requires less thermal energy to create movement between molecules. Methacrylates substitute a larger methyl group for the hydrogen atom, which restricts the polymer's freedom to rotate and thus gives the methacrylate a higher T_g.

Figure 3: Acrylic Monomer Units[64]



The hindrance in movement from the larger methyl group also gives methacrylate polymers a higher tensile strength and a lower elongation percentage than the acrylate polymers. The length of the alkyl ester side chains affect the properties of the polymers; as the alkyl ester side chains become

[64] "Polyacrylates," *The Polymer Science Learning Center*, http://pslc.ws/macrog/acrylate.htm.

longer, the tensile strength decreases and the elongation increases.[65] As seen in Table 2, for both acrylates and methacrylates, at room temperature the polymer of methyl is tougher and less pliable than the polymers of n-butyl. In general, the softness of the polymers increases as the length of the alkyl group chain increases.[66] And finally, the influence of the ester side group in enhancing the polymer overall chain stiffness depends on the flexibility and bulkiness of the ester group. Side groups that are fairly bulky limit how closely the polymer chains can pack together. The further they are from each other, the more easily they can move around, which in turn increases the free volume, and consequently decreases the T_g. This effect, shown in Table 2, is demonstrated in both the polyacrylate and polymethacrylate series where the increased distance between the chains, reduces the T_g. The methacrylate maintains higher overall T_gs due to the larger methyl group described above.

Table 2: Decrease of T_g with Increasing Flexibility of Side Chains in the Polyacrylate and Polymethacrylate Series [67]

Ester Alkyl Side Chain	Acrylate - Tg °C	Methacrylate - Tg °C
Methyl	9	105
Ethyl	-22	65
Butyl	-54	20

[65] David R Gehman, "Acrylic Adhesives," *Handbook of Adhesives* (New York: Chapman & Hall, 1990), 439.

[66] *Synthetic Resins* 53.

[67] Gehman 440.; "The Glass Transition," *The Polymer Learning Center*. http://pslc.ws/macrog/tg.htm.

Since the T_g of an adhesive is lower when there is an increase in free volume, low molecular weight additives such as plasticizers, solvents, water, or adhesives with lower T_g values are often used to separate the molecular chains, thereby creating less resistance to movement.[68, 69] Plasticizers decrease the T_g of the cured adhesive by decreasing the molecular weight and increase the flexibility of the adhesive. While solvents and water also decrease the T_g, once the solvent or water evaporates during the curing process, the T_g will once again increase. If solvent remains in the adhesive after setting, the T_g will continue to be lower and the film will be more flexible. Solvent typically stays in the film when the T_g of the adhesive is greater than the ambient temperature. According to Michael Schilling's study on the T_g of materials used in conservation, it is best to choose an adhesive with a T_g that is lower than the ambient temperature in order to drive out the solvent entirely, ultimately creating a much harder adhesive and achieving the maximum T_g of the material.[70] However, for conservators, this is not a viable option because the higher ambient temperature would mean that the adhesive would always be soft. The ideal situation would be to heat the object with the adhesive while it cures so that the solvent can fully evaporate before returning the object to a temperature below the T_g of the adhesive. Again, this is almost never an option for a conservator.

C. Thermal Expansion Coefficient

When acrylic polymers are above their T_g, the molecular chains begin to rotate, causing an increase in the free-volume. In other words, the polymeric material expands and swells, and as a result, the linear coefficient of thermal expansion (LCTE) and elasticity increase. The LCTE of a polymeric material is three to five times greater when it is above the T_g. Drastic dimensional changes such as this can cause severe problems in certain circumstances. For example, consider an adhesive

[68] Horie 25.

[69] Stephen Koob also suggests that the addition of fumed colloidal silica helps with all the rheological properties of linear polymers. Cf Koob (1986): 9.

[70] Schilling 111-112.

that is applied to a material with a lower LCTE. The material and the adhesive joint are exposed to normal temperature cycles of the surrounding environment. If those temperature cycles extend above and below the T_g of the polymer, large dimensional changes will occur in the adhesive as it cycles between the lower LCTE value (below the T_g) and the higher LCTE value (above the T_g). For materials with a relatively low, constant LCTE, the stresses induced by the cycling may cause the adhesive to crack and break. Therefore, when choosing an adhesive, the T_g should not be close to ambient temperatures, since dramatic dimensional changes could occur when the temperature fluctuates around the T_g. This is especially important if the adhesive and the substrate expand and contract to different degrees.[71] Since significant differences in LCTE can cause bond failure and damage the substrate, use of a low modulus, flexible adhesive can help reduce thermal stresses between the adhesive and the substrate.[72]

D. Deterioration of Polymers

As expressed by Velson Horie, "polymers used in conservation must not change and cause harm to the objects."[73] Conservators understand that these organic materials will not last forever, but the expectation is for an adhesive is the always remain fully reversible without causing any damage to an object. While complete reversibility is still the ultimate goal, it is more likely that some aspects of the treatments will never be fully reversible. With that acknowledgement, it is important that conservators choose materials that have long-term stability to allow removal of prior treatments and subsequent re-treatment of an object.[74]

Degradation of polymer resins leads to yellowing, increased brittleness, loss of strength or solubility, shrinking, flowing, dirt accretions, and physical or chemical reactions with the substrate.

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71 Schilling 111.

72 Down 22-24.

73 Horie 37.

74 ibid 37.

The three primary causes of these changes are light, heat, and oxygen, though the long-term performance is mainly influenced by the direct action of sunlight in promoting oxidation reactions. The effects of oxidation may be accelerated by temperature, moisture, and atmospheric pollutants. Since adhesives often fail due to deterioration of the polymers, it is important to understand the effects of aging (i.e. light, heat, and oxygen) on adhesives. Aging studies can include thermal, photochemical, humidity cycling, or exposure to pollution, and testing typically involves measuring the yellowing, solubility, strength, and pH before and after aging.[75]

Thermal degradation is caused by high temperatures breaking the molecular chains of the polymers either by cross-linking or by chain scissions. Polymers that become cross-linked are insoluble because the polymer chains react with each other to form an intractable three-dimension network. Chain scissions, which is the alternative to cross-linking, is when the polymer chains are simply broken. The molecular weight is halved for each break and the polymer is considerably weaker.[76] The two reactions occur relatively independently and their rates respond differently to changes in the deteriorating environment. During degradation, when a polymer only reacts by chain scission it remains permanently soluble, becoming weaker and eventually liquefying. For cross-linking, each time a polymer cross-links, the molecules increase in size forming an insoluble network.[77] In 1977, when Feller was studying the photochemical degradation of polymers, he noticed a different degree of cross-linking above and below the T_g of poly(isobutyl methacrylate). Above the T_g cross-linking occurred exclusively; below the T_g considerable chain scissions occurred.[78] He followed up on this study years later by observing the behavior of poly(vinyl butyral) (Butvar B-79) when exposed to temperatures above and below its T_g (63 °C). Feller found that cross-linking took place exclusively at temperatures high above the T_g but at temperatures below the T_g

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75 Down 25; Chiantore et al (2001) 17.

76 Horie 42.

77 ibid 43.

78 Feller (1981) 192.

considerable chain scission occurred. However, the cross-linking was not a sharp "on and off" behavior; there were intermediate degrees of chain scission to cross-linking observed at temperatures only slightly below the T_g. Feller's explanation is that above the T_g, the polymer molecules, particularly the ester side chain groups, are in considerable thermal motion; therefore, if a free radical (unshared electron) site is generated at some point in the polymer structure, the movement of the molecules provides the opportunity to link up with a neighboring chain. However, at temperatures below the T_g, molecular motion is "frozen" providing no opportunity for cross-links to develop between neighboring chains.[79]

In more recent tests on acrylic consolidants, researchers confirmed that the stability of the resins appears to be controlled by the reactivity of the alkyl side groups. Oxidation, which is the reaction of oxygen with radicals formed in the polymer, typically occurs in the side groups, favoring long esters such as resins containing butyl groups, and causes the adhesives to undergo fast and extensive cross-linking. However, research has shown that B-48N, which contains a small amount of BMA units, does not form anhydrides, which are chemical compounds formed by the elimination of water, due to the presence of the more stable co-units. In contrast to the resins containing butyl, only a limited amount of decomposition of the esters occurred in B-44 (methyl methacrylate and ethyl acrylate) and B-72 (ethyl methacrylate and methyl acrylate). These findings indicate that both the acrylic and methacrylic resins, where either all or most the alkyl side groups are short, experience chain scission more often than cross-linking and no insoluble fractions are formed.[80]

Photochemical degradation is the result of polymers absorbing photons when exposed to light. The absorbed energy causes the chemical bonds in the polymer's backbone to break apart creating free radicals, which can then react further with the oxygen in the atmosphere (i.e. photo-oxidation). The energy absorbed from ultraviolet (UV) radiation is enough to break the majority of the chemical bonds, however, over an extended amount of time, the energy absorbed from the visible

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[79] R.L. Feller, "Thoughts about Crosslinking," *WAAC Newsletter* Vol 30 No 3 (2008): 16-17.

[80] M. Lazzari, O. Chiantore, "Thermal-Aging of Paraloid Acrylic Protective Polymers," *Polymer* 41 (2000):6454-6455.

portion of the spectrum will also cause chain scissions.[81] In 1981, Feller published a study on the photochemical stability of methacrylate coatings. Using accelerated-aging tests involving a carbon-arc radiometer at high levels of UV light, he found that the higher alkyl methacrylates (such as those containing butyl) and all of the acrylates cross-link under UV radiation, though the acrylate units are more reactive towards oxidation compared with the methacrylates. In both units, when the ester side group is short, scission reactions are more common than cross-linking. However, the methyl and ethyl groups do not cross-link as often as amyl, butyl, and propyl groups. With a butyl ester group, the behavior of the polymer changes dramatically, undergoing fast and extensive cross-linking even in the case of irradiation at longer wavelengths, such as visible light. Therefore, Paraloid B-72, which is an ethyl methacrylate/methyl acrylate copolymer tends to chain break, rather than cross-link, under visible and near UV radiation, although at a very slow rate.[82] This is because the methyl group prevents a reaction with oxygen and therefore the polymer does not cross-link. Poly(methyl methacrylate) (PMMA) under accelerated conditions requires even longer irradiation times before any photo-induced oxidation is detected.[83]

[81] Horie 39-40.

[82] Feller et al. (1981) 184-193.; Chiantore et al. (2001) 18.

[83] Feller et al (1981) 187.

SECTION 2: MATERIALS

2.1 Adhesives Used in this Study

Four different Paraloid acrylic resin adhesives, and two composite systems, are used in this study: B-72, B-48N, B-44, A-11, 3:1 B-72:B-48N, 1:3 B-72:B-48N. As discussed, these resins, due to their relatively low glass transition temperatures (with the exception of A-11), are currently advisable for indoor use only; however, interestingly there are many instances of outdoor use beginning in the 1960s.[84] The B-72, B-48N, and B-44 adhesives were produced as 50% solutions made in the following manner: 50g Paraloid, 50g acetone.[85] The A-11 adhesive was produced as a 40% solution made in the following manner: 40g A-11, 60g methyl ethyl ketone.[86] The density of MEK is slightly higher than that of acetone (MEK density = 0.8049 g/mL at 20° C and acetone density = 0.7900 g/mL at 20° C) which was used for all of the other adhesives. A-11 is soluble in ethylene dichloride, Cellosolve® acetate, ethyl acetate, toluene, DMF, MEK. MEK was chosen because its vapor pressure (74 Torr at 20° C) is closest to that of acetone (184.5 Torr).[87] The mixtures of B-72 and B-48N were created from the 50% solutions in a 3:1 and 1:3 mixture (by volume).

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[85] J.M. Cabrera Garrido, "The Portal of the Monastery of Santa Maria de Ripoll," Monumentum 1 (1967): 79-98.; P. Mora et al., "Consolidamento Provvisorio di un Marmo in Stato de Avanzata Degradazione*," Proceedings of the International Meeting*, La Conservazione delle sculture all'aperto, Ente Bolognese Manifestazioni Artistiche, Bologna (1969): 160-62.; Ottorino Nonfarmale, "A Method of Consolidation and Restoration for Decayed Sandstone," *The Conservation of Stone I*. Proceedings of the International Symposium, Bologna, June 19-21 (Bologna: Centro per la conservazione delle sculture all'aperto, 1975).; R. Rossi-Manaresi, "Treatments for Sandstone Consolidation," *The Conservation of Stone I*. Proceedings of the International Symposium, Bologna, June 19-21 (Bologna: Centro per la conservazione delle sculture all'aperto, 1975).; Thomas C. Roby, "In Situ Assessment of Surface Consolidation and Protection Treatments of Marble Monuments in Rome of the 1980's, with Particular Reference to Two Treatments with Paraloid B-72," *Proceedings from the Eight International Congress on Deterioration and Conservation of Stone, Berlin, 30 Sept - 4 Oct. 1996* (Berlin: Möller Druck und Verlag, 1996): 1015-1028.

[85] Volume for 50g of acetone = 63.2 mL

[86] Volume for 50g of MEK = 62.1mL

[87] MEK also has a higher vapor pressure than ethanol (45 Torr at 20°C) which is often used along with acetone in these adhesive mixtures. "Vapor Pressure," http://macro.lsu.edu/HowTo/solvents/Vapor%20Pressure.htm. - note that it's higher than ethanol and put ethanol.

Table 3: Adhesive Properties [88, 89]

	Polymer/ Copolymers	Chemical Composition	Composition of the Resin (mol%)	Tg (Reported)	Molecular Weight (Wt. Avg.)	Solubility Parameter	Refractive Index	Ultimate Hardness of Clear Films, KHN
Paraloid B-72	ethyl methacrylate and methyl acrylate	EMA Copolymer (EMA/MA)	32.0 MA 65.8 EMA 2.2 BMA	40°C	105,000	9.3	1.47-1.48	10-11
Paraloid B-48N	methyl methacrylate and butyl acrylate	MMA Copolymer (MMA/BA)	74.5 MMA 25.5 BMA	50°C	250,000	9.3	1.89	11-12
Paraloid B-44	methyl methacrylate and ethyl acrylate	MMA Copolymer (MMA/EA)	about 28 EA; 70.3 MMA	60°C	140,000	9.4	1.47	15-16
Paraloid A-11	methyl methacrylate	MMA Polymer	100 MMA	100°C	no data	9.4		18-19

2.2 Description of the Indiana Limestone

Limestone is sedimentary rock composed mainly of calcium carbonate with a range in grain-size, color, and impurities. Formed in marine environments, limestone is created from both deposits of chemical precipitation and from the consolidation of calcareous marine fossils. Typically limestone, due to the nature of its formation, exhibits a few distinguishing features such as calcite

88 Paraloid™ B-72 (50%) Thermoplastic Solution Resin , 2007 Technical Data Sheet, Rohm and Haas, accessed April 9, 2017, http://www.palmerholland.com/Assets/User/Documents/Product/42468/504/MITM04034.pdf.; PARALOID™ B-48N 100% Solid Grade Thermoplastic Acrylic Resin, 1996 Technical Data Sheet, Rohm and Haas, accessed April 9, 2017, http://www.dow.com/assets/attachments/business/pcm/paraloid_b/paraloid_b-48n_100_pct/tds/paraloid_b-48n_100_pct.pdf.; PARALOID™ B-44 100% Solid Grade Thermoplastic Acrylic Resin, 1996 Technical Data Sheet, Rohm and Haas, accessed April 9, 2017, http://www.dow.com/assets/attachments/business/pcm/paraloid_b/paraloid_b44/tds/paraloid_b-44_100_pct.pdf.; PARALOID™ A-11 Solid Grade Thermoplastic Acrylic Resin, 1996 Technical Data Sheet, Rohm and Haas, accessed April 9, 2017, http://www.palmerholland.com/Assets/User/Documents/Product/42429/498/MITM03991.pdf.; "Technical Properties of Paraloid® Resins," CAMEO: Conservation and Art Materials Encyclopedia (Museum of Fine Arts, Boston, 2007) http://cameo.mfa.org/images/1/12/Download_file_539.pdf.

89 There is conflicting literature on the exact numbers for these resins. Recent literature on B-72, which is considered a binary copolymers of methyl acrylate/ethyl methacrylate (MA/EMA), by O. Chiantore, M. Lazzari found samples of B72 contained a low amount of butyl methacrylate, (BMA), units. Cf O. Chiantore, M. Lazzari, "Photo-oxidative stability of Paraloid acrylic protective polymers," *Polymer* 42 (2001): 17–27.; A chart showing the various numbers reported for these adhesives can also be found in Horie's *Materials for Conservation* on pg 160.

streaks, fossils or shell formations, pit holes, honeycomb formations, or iron spots.[90] Indiana limestone, also known as Bedford Oolitic or Salem Limestone, has a high percentage of calcium carbonate, is characterized by its warm, neutral-colored, and concentric rings of $CaCO_3$, which forms around the grains. Most of the constituents that make up Indiana limestone are sand-sized, fossilized debris of marine organisms. The fossil debris is bound together by calcite cement, or lime mud known as micrite. Optically clear calcite, known as spar, occurs throughout Indiana Limestone. The micrite has an average diameter of ~5 μm, whereas the sparry grains are coarser, with an average diameter of 25 μm.[91] While subtle color and grain differences are present, Indiana limestone is extremely homogenous making it well-suited for buildings, monuments, and sculptures.[92]

Indiana limestone has long been considered one of the most durable stones due to its uniformity in composition and texture and ability to withstand extreme heat and cold. Even before Indiana was admitted into the Union in 1816, pioneers were using Indiana's limestone for architecture and monuments. With the introduction of the railroads and the devastating fires in Chicago (1871) and Boston (1872), demand for Indiana limestone increased rapidly. In 1876, Indiana Stonework won awards of merit for quality at both the Philadelphia and New Orleans Centennial Expositions, and by the end of the 19th century, demand for Indiana limestone was so great that the number of quarries doubled between 1889 and 1895. During the early 20th century, technological advancements allowed architects and sculptors to use the material to fit the changing styles of the Art Deco period. The fine, uniform grain of Indiana limestone allowed artists and architects to create new and interesting surface textures by carving into the surface of the stone. [93]

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[90] "Color Grades," Indiana Limestone Institute of America, 2017, http://www.iliai.com/pages/Colors_Grades.

[91] Wei Shu, Patrick Baud, and Teng-fong Wong, "Micromechanics of Cataclastic Pore Collapse in Limestone," *Journal of Geophysical Research*, Vol. 115 (2010): 5.

[92] "Advantages," Indiana Limestone Institute of America, 2017, http://www.iliai.com/pages/Advantages.

[93] "History," Indiana Limestone Institute of America, 2017, http://www.iliai.com/pages/History.

2.3 Description of the Terracotta

The terracotta used in this thesis is a modern yellow iron spotted brick provided by Highbridge Materials Consulting Inc. Terracotta is an important and versatile ceramic material that has been used by humans in a variety of ways since prehistoric times.[94] From sculpture, to pottery, to tableware, to architecture, almost every ancient civilization created works of beauty using terracotta.[95] Ancient terracotta is the simplest and coarsest type of pottery — it is lightweight, very porous, and typically created from a red-colored clay that is fired at temperatures below 850° C. The low firing temperatures result in only slight vitrification and consequently a highly porous material.[96]

Modern architectural terracotta became popular in the mid-1800s as a lightweight fireproofing material for building facades and ornamental elements. Modern terracotta is manufactured from high quality clay, primary 60-70% silicon dioxide and 20-30% aluminum oxide, which forms a cementing or consolidating matrix as the clays are heated. Clay is mined from the ground and then allowed to weather age before use. Weathering increases the clays' plasticity and converts sulfides in the clay into sulfates and oxides through oxidation. Clays with different chemical compositions can be blended together to created specific mixtures with a particular set of characteristics called a clay body. Once the clay has dried out sufficiently, it is washed to remove impurities and then mixed with grog which is typically very fine white sand, pulverized firebrick, fragments of ceramic material. The clay is then molded and fired at temperatures reaching 1205° - 1927° C.[97] While the higher firing temperatures increase vitrification slightly, modern architectural terracotta is still a highly porous and opaque ceramic material.

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[94] Venus of Dolni Vestonice (26,000-24,000 BCE)

[95] T. Ashby, "Terra-Cotta in Archaic Art," *The Classical Review* 38 No. 3/4 (1924): 76-77.; Jane Portal, *Terracotta Warriors: Guardians of China's first Emperor* (Washington D.C.: National Geographic, 2008).; Karl-Ferdinand Schädler*, Earth and Ore: 2500 Years of African Art in Terracotta and Metal* (Eurasburg: Edition Minerva München Distribution, Panterra Verlag, 1997).; Arputha Rani Sengupta, *Art of Terracotta: Cult and Cultural Synthesis in India* (Delhi: Agam Kala Prakashan, 2005).; J.F. Jemkur, *The Nok Culture: Art in Nigeria 2,500 Years Ago* (Munich and New York: Prestel 2006).

[96] Zvi Goffer, *Archeological Chemistry* (New Jersey and Canada: John Wiley & Sons, 2007) 247.

[97] Richard Veit, "Moving Beyond the Factory Gates: The Industrial Archaeology of New Jersey's Terra Cotta Industry," *Journal of the Society for Industrial Archeology* (1999): 7-10.

SECTION 3: EXPERIMENTAL

3.1 Experimental Design

This thesis involves the use of the adhesives discussed in the previous chapter, in bonding drilled cores of limestone and terracotta. For each adhesive, sixty specimens are prepared — 30 limestone and 30 terracotta. The bonded specimens are then divided into two testing groups and one control group. Group A is subjected to thermal cycling between 30-60° C, followed by four-point bend flexure testing to determine the stiffness, bond strength, and mode of failure of the adhesives.

For Group A, mechanical properties are determined using a four-point bend accessory. From the resulting force-displacement graph the flexural strength and flexural modulus are calculated and the mode of failure revealed. Thermoplastic materials that are below their T_g have higher flexural moduli. Above their T_g, some thermoplastics begin to flow immediately when force is applied; as the stretching continues, the polymers eventually distort irreversibly at the yield point. The distortion will continue with further stretching until the material finally breaks at the point of rupture. If the polymer is below its T_g or heavily cross-linked there will be little distortion before the breaking point.[98]

Group B examines the thermally induced creep behavior of the adhesives. Each sample rests on two supports and is heated with a infrared lamp while under a constant load. The overall configuration mimics a four-point bend test. Each specimen is timed from the moment the UV lamp is turned on (the weight is applied before the lamp is turned on) until the joint fails. Temperatures are monitored with an infrared thermometer to determine the correlation between temperature and bond failure.

[98] Horie 28-30.

A. Sample Preparation

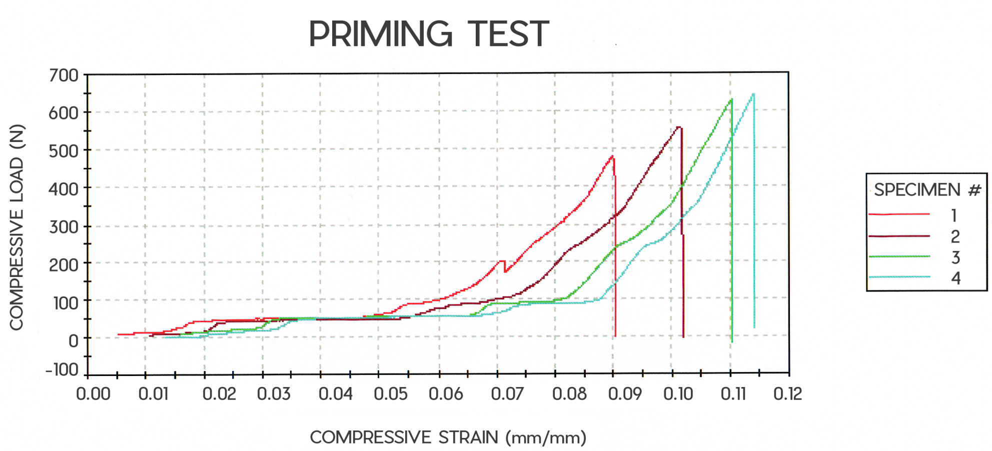
The limestone core specimens were drilled from a block of Indian Limestone using a Delta drill press, with a water swivel 5/8 - 18 thread and 1/2" diameter shank adapter attached to 1/2" diameter diamond core drill bit. The cores were then left to air-dry for four weeks before being dried in an oven for 48 hours at 60° C. The final limestone samples were cylinders with a diameter of 16.61 mm and a height of 78.40 mm. Using an Instron 4201, the limestone cores were then subjected to incremental increasing pressure until failure. This process created consistently fractured surfaces through the middle of the specimens. In preparation for the adhesive, the fractured surfaces were brushed using a synthetic Loew-Cornell 1136 brush in order to remove dust and debris.

The terracotta samples were drilled from a number of blocks provided by Highbridge Materials Consulting, using a Delta laser drill press, with a water swivel 5/8 - 18 thread and 1/2" diameter shank adapter attached to 1/2" diameter diamond core drill bit. The cores were then left to air dry for four weeks before being dried in an oven for 48 hours at 60°C. Due to the variation in the terracotta from block to block, the cores needed to be trimmed down to ensure that all of the specimens were the exact same length and would, therefore, fit inside of the brackets while the adhesives set. The specimens were cut using a Covington Engineering tile saw. The cores were then re-dried in the oven for 48 hours at 60°C. The final terracotta samples were cylinders with a diameter of 16.61 mm and a height of 92.65 mm. Using an Instron 4201, the terracotta cores were then subjected to incremental increasing pressure until breaking. This process created consistently fractured surfaces through the middle of the specimens. In preparation for the adhesive, the fractured surfaces were brushed using a synthetic Loew-Cornell 1136 brush in order to remove dust and debris.

B. Application of the Adhesive

Prior to applying adhesive to the terracotta specimens, testing was performed to determine to what extent a priming coat would affect the strength of the adhesive joint. Terracotta is a porous material, and without a barrier layer, the adhesive would potentially be drawn into the substrate, away from the surface, creating a weak bond at the joint, resulting in inaccurate measurements during testing.[99] Three specimens were prepared: 1) no priming coat, bonded with 50% B-72 solution; 2) 10% B-72 (w/v) in acetone priming coat, bonded with 50% B-72 solution 2) 20% B-72 (w/v) in acetone prime coat, bonded with 50% B-72 solution. After setting for two weeks, the specimens were subjected to four-point bend testing on the Instron 4201. As seen in Figure 4, the specimen with the 20% prime coat (specimen 4) had a higher ultimate strength, indicating that the prime coat had sufficiently penetrated the substrate and increased the overall strength of the adhesive joint. As a result, a 20% B-72 (w/v) in acetone solution was applied by brush to each half cylinder of terracotta, covering the fractured surfaces. The barrier coat was then left to set at room temperature for three days.

Figure 4: Priming Layer Test for Terracotta



[99] A priming layer was on the terracotta because of its high permeability but it was not used on the limestone due to its low permeability.

The six adhesives were applied to the limestone and terracotta specimens using a #6 Red Sable 7500 Series round brush dipped into the container holding the solution.[100] Excess adhesive was brushed away against the rim of the container. The adhesive was then brushed onto the fractured surfaces of both halves, covering them entirely, before the two halves were then carefully attached back together. The halves were pressed together firmly to ensure good contact and adhesion and then placed in a clamp where they set for five weeks. With the exception of the A-11 specimens, excess adhesive around the circumference of the joint was left to set and then removed mechanically with a scalpel during the third week of setting.

C. Setting of the Adhesive

During the reconstruction of the Tullio Lombardo's *Adam*, Riccardelli and Jorjani developed a sample preparation protocol for applying the optimal pressure needed for maximum adhesion. They conducted an empirical test using a layer of 40% B-72 in acetone solution (by weight) applied to marble with pressures of 50, 100, and 200 psi. Their results showed that at 100 psi there was enough adhesive remaining in the joint so as not to create a "dry joint," but not so thick a layer as to obstruct the adhesion by allowing the marble cubes to slide off one another.[101] [102]

The clamping system used was also based on the design created by Riccardelli and Jorjani. The clamps were built using two pieces of plywood measuring 16" x 1"x 0.75", connected with a series of six 6" bolts for the terracotta, 5" bolts for the limestone, and secured with 1/4" nuts (Figure 5). The holes to accommodate the bolts were spaced 3" inches apart to accommodate ten glued

[100] A limitation of using a brush, is that the open jar allows for solvent evaporation, thereby changing the viscosity of the mix. Approximately 0.5 gram of acetone is lost per minute out of an open jar.

[101] Jorjani 22.

[102] This thesis only analyzed specimens that were clamped at 100psi in order to ensure consistency among the samples. In future studies, it would be beneficial to expand the research to include specimens that were clamped at a constant pressure as well as those held together by hand as this is closer to how these adhesives are being applied in the field.

specimens. Once all ten specimens were bonded, they were placed into the clamp and the bolts were tightened to approximately 100 psi using a torque wrench.

Figure 5: Clamping System

The transition from pressure to torque was calculated using the following equation:

$$\text{Moment} = (W^*p)/(2\pi^* e)$$

In this equation, the calculation for moment allows us to use a torque wrench to achieve the optimal pressure of 100 psi. Here, W is the work done by the lever, p is the screw pitch or threading, and e is the efficiency of the clamps.[103] For our purposes:

[103] Jorjani 23.

100 psi of pressure

16 in. of clamping area

6 bolts per clamp

To determine W, or work:

16 in. x 100 psi = 1600 lb. of pressure

1600 lb.. / 6 (screws) = 266.7 lb. of force/screw

W = 266.7 lb.

To determine p, or pitch:

Screw threads = 1/4 - 20

p = 0.050 in.

Efficiency, e, is an assumed constant used by Jorjani.[104]

e = 0.25

$$\text{Moment} = (266.7 \text{ lb.} * 0.050) / (2\pi * 0.25) = 8.48 \text{ in-lb.}$$

All of the bolts on each clamp were tightened to 8.5 in-lb. using a torque wrench (the torque wrench measurements are in increments of 1 in-lb.). This ensured that a pressure of approximately 100 psi was applied to each cylinder during the setting process. The bonded cylinders were allowed to set under pressure for six and half weeks at room temperature. The specimens continued to set after they were unclamped until they were subjected to either the four-point testing or the thermal creep

[104] ibid 23.

testing. See Figure 8 below for the start and end dates of the curing as well as the test day for each adhesive and specimen.

Once the adhesives were done setting, the specimens were divided into Group A (thermal cycling and four-point bend), Group B (stress-creep behavior), and Group C (control group). As shown in Appendix 2, Group A included 140 specimens total — 60 limestone with 10 from each of the 6 adhesives; 60 terracotta with 10 from each of the 6 adhesives; 20 control specimens including 10 unbroken limestone and 10 unbroken terracotta. Group C, listed in Appendix 3, was the control group for Group A and included 120 specimens total — 60 limestone with 10 from each of the 6 adhesives; 60 terracotta with 10 from each of the 6 adhesives. Group B, shown in Appendix 4, also included 140 specimens total — 60 limestone with 10 from each of the 6 adhesives; 60 terracotta with 10 from each of the 6 adhesives; 20 control specimens including 10 unbroken limestone and 10 unbroken terracotta. All 280 specimens were photographed and put through an empirical "roll test" prior to any thermal testing. The roll test is an empirical analysis used to determine whether or not the adhesive had changed in size or shape between each testing phase.

Table 4: Limestone and Terracotta Groups

LIMESTONE

Group A	B-72	Limestone_B-72_01-10
	B-48N	Limestone_B-48N_01-10
	B-44	Limestone_B-44_01-10
	A-11	Limestone_A-11_01-10
	1:3 B-72:B-48N	Limestone_1B-72_3B-48N_01-10
	3:1 B-72:B48N	Limestone_3B-72_1B-48N_01-10
	unbroken, cycled	Limestone_CT_NA_01-10
Group B	B-72	Limestone_B-72_11-20
	B-48N	Limestone_B-48N_11-20
	B-44	Limestone_B-44_11-20

	A-11	Limestone_A-11_11-20
	1:3 B-72:B-48N	Limestone_1B-72_3B-48N_11-20
	3:1 B-72:B48N	Limestone_3B-72_1B-48N_11-20
	unbroken	Limestone_CT_NA_11-20
Group C (control)	B-72	Limestone_B-72_21-30
	B-48N	Limestone_B-48N_21-30
	B-44	Limestone_B-44_21-30
	A-11	Limestone_A-11_21-30
	1:3 B-72:B-48N	Limestone_1B-72_3B-48N_21-30
	3:1 B-72:B48N	Limestone_3B-72_1B-48N_21-30
	unbroken, uncycled	Limestone_CT_NH_21-30

TERRACOTTA

Group A	B-72	TerraCotta_B-72_01-10
	B-48N	TerraCotta_B-48N_01-10
	B-44	TerraCotta_B-44_01-10
	A-11	TerraCotta_A-11_01-10
	1:3 B-72:B-48N	TerraCotta_1B-72_3B-48N_01-10
	3:1 B-72:B48N	TerraCotta_3B-72_1B-48N_01-10
	unbroken, cycled	TerraCotta_CT_NA_01-10
Group B	B-72	TerraCotta_B-72_11-20
	B-48N	TerraCotta_B-48N_11-20
	B-44	TerraCotta_B-44_11-20
	A-11	TerraCotta_A-11_11-20
	1:3 B-72:B-48N	TerraCotta_1B-72_3B-48N_11-20
	3:1 B-72:B48N	TerraCotta_3B-72_1B-48N_11-20
	unbroken	TerraCotta_CT_NA_11-20
Group C (control)	B-72	TerraCotta_B-72_21-30

	B-48N	TerraCotta_B-48N_21-30
	B-44	TerraCotta_B-44_21-30
	A-11	TerraCotta_A-11_21-30
	1:3 B-72:B-48N	TerraCotta_1B-72_3B-48N_21-30
	3:1 B-72:B48N	TerraCotta_3B-72_1B-48N_21-30
	unbroken, uncycled	TerraCotta_CT_NH_21-30

3.2 Experimental Testing — Group A

Table 5: Summary of Appendix 2 for Group A

Substrate and Adhesive	Bonding Start Date	Bonding End Date	Thermal Cycling Start Date	Thermal Cycling End Date
Limestone_B-72_01-10	2/3/2017	3/13/2017	3/17/2017	4/3/2017
Limestone_B-44_01-10	2/3/2017	3/13/2017	3/17/2017	4/3/2017
Limestone_B-48N_01-10	2/3/2017	3/13/2017	3/17/2017	4/3/2017
Limestone_1B-72_3B-48N_01-10	2/3/2017	3/13/2017	3/17/2017	4/3/2017
Limestone_3B-72_1B-48N_01-10	2/3/2017	3/13/2017	3/17/2017	4/3/2017
Limeston_A-11_01-10	2/3/2017	3/13/2017	3/17/2017	4/3/2017
Limestone_Control_ NA_01-10 (no adhesive)	N/A	N/A	3/17/2017	4/3/2017
TerraCotta_B-72_01-10	2/1/2017	3/13/2017	3/17/2017	4/3/2017
TerraCotta_B-44_01-10	2/2/2017	3/13/2017	3/17/2017	4/3/2017
TerraCotta_B-48N_01-10	2/2/2017	3/13/2017	3/17/2017	4/3/2017
TerraCotta_1B-72_3B-48N_01-10	2/2/2017	3/13/2017	3/17/2017	4/3/2017
TerraCotta_3B-72_1B-48N_01-10	2/2/2017	3/13/2017	3/17/2017	4/3/2017
TerraCotta_A-11_01-10	2/2/2017	3/13/2017	3/17/2017	4/3/2017
TerraCotta_Control_NA_01-10 (no adhesive)	N/A	N/A	3/17/2017	4/3/2017

A. Thermal Cycling

Group A specimens were placed horizontally in a Humboldt Bench Series Ovens Model 51 ER environmental chamber and cycled between temperatures ranging from 30-60° C for one week. The oven is run by a CAL 9500P programmable process controller. Within each 24-hour period the specimens underwent 8 complete cycles. The limitation for environmental cycling is the amount of time that it takes for each core to cool down to ambient temperature. After thermal cycling was complete, a second roll test was conducted in order to determine if the elevated temperatures caused any changes to the specimen's shape or size. The highest temperature, 60° C is higher than the glass transition temperature of all the adhesives tested, except for Paraloid B-44 (which has a T_g of 60° C) and Paraloid A-11 (which has a T_g of 100° C).

B. Four-Point Bend Flexure Testing

Following thermal cycling, four-point bend flexure testing was conducted using an Instron 4467 mechanical strength analyzer. The Instron 4467 is controlled by a proprietary software entitled Bluehill Software 2 version 2.4. The Instron graphs the load (N) versus displacement (mm) for each specimen. Flexure testing is carried out at room temperature. The load is applied with a 5 kN load cell at a rate of 0.01 mm/sec until the critical load is reached. The data are recorded by the Instron 4467 and a graph is generated for each individual specimen. The graphs show the relationship between the load (or force) and the amount of deformation of the adhesive specimens prior to failure.

C. Results and Discussion

Force (Stress) vs. Displacement graphs for all of the samples in Group A are found in Appendix 2.

Limestone

For all of the limestone groups there is a low standard deviation, especially as compared to the high standard deviation observed in the terracotta specimens. This is due to the heterogeneity of terracotta versus the uniformity of limestone. In general, results indicate that all of the adhesive systems had a lower strength than the unbroken control group (L_CT_NA) and the unbroken, uncycled control group (L_CT_NH).

Table 6: Percent Difference in the Limestone Samples

Substrate and Adhesive	Average Strength (MPa)	Standard Deviation	% Difference from Control Cycled	% Difference from Control Uncycled
L_B72	6.53	0.88	-18.94	-11.02
L_B48N	6.21	0.52	-22.89	-15.36
L_B44	6.57	1.00	-18.46	-10.50
L_A11	6.09	1.03	-24.43	-17.05
L_1B72_3B48N	6.42	0.67	-20.33	-12.54
L_3B72_1B48N	6.67	0.61	-17.24	-9.15
L_CT_NA (control cycled)	8.06	0.28	N/A	9.77
L_CT_NH (Group C- control uncycled)	7.34	0.36	-8.90	N/A

For the limestone, four-point bend flexure testing results in similar values for all adhesives. In every case the mode of failure was brittle, though the fracture occurred most often in the stone, next to the adhesive joint. As seen in Table 6, the range in average strength is very low — the 3:1

B-72:N-48N mixture has the highest average strength at 6.670 MPa and A-11 has the lowest at 6.090 MPa. This small range, observed only in the limestone, is both good and surprising, as it indicates that even B-72 retains a significant amount of strength after 100 temperature cycles ranging from 30-60°C. While A-11, the adhesive with the highest reported T_g, performed the worst, overall, the adhesives tested performed almost equally with the limestone substrate.

Terracotta

For each adhesive system, all of the bonded specimens, with the exception of the B-72 specimens, were stronger than the unbroken control groups. B-72, which has the lowest T_g, performed below all of the other adhesive systems, however when compared to the control groups, B-72 performed better than the unbroken, uncycled. This indicates that the adhesives are creating a joint that is on average greater than the strength of the material, but not significantly greater as to potentially cause damage to the substrate.[105] For the purposes of conservation, all of these adhesives performed very well and would not be considered too strong, as to potentially cause damage to the ceramic material. In general, the adhesive systems perform better with the terracotta than with the limestone. There was an overall decrease in strength for every adhesive system when compared to both control groups (L_CT_ NA and L_CT_NH) in the limestone material, but an overall increase in strength of the adhesive systems compared to the uncycled (TC_CT_NH) terracotta control group.[106]

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[105] These are similar results to those reported by Jorjani in 2009.

[106] By some theories of adhesion, these resins should be more compatible with terracotta than with calcite due to the acid base nature of the substrate versus the adhesive. Cf. F. Fowkes, "Role of acid-base interfacial bonding in adhesion," *Journal of Adhesion Science and Technology* (1987) 7-27.

Table 7: Percent Difference in the Terracotta Samples

Substrate and Adhesive	Average Strength (MPa)	Standard Deviation	% Difference from Control Cycled	% Difference from Control Uncycled
TC_B72	10.08	2.23	-6.81	9.00
TC_B48N	11.79	2.56	8.67	22.19
TC_B44	13.09	3.68	17.76	29.94
TC_A11	11.40	2.00	5.60	19.58
TC_1B72_1B48N	12.22	2.40	11.92	24.95
TC_3B72_1B48N	10.69	2.89	-0.73	14.18
TC_NA (control cycled)	10.76	3.69	N/A	14.80
TC_CT_NH (Group C- control uncycled)	9.17	3.62	-17.37	N/A

3.3 Experimental Testing — Group C

Table 8: Summary of Appendix 3 for Group C

Substrate and Adhesive	Bonding Start Date	Bonding End Date	Four-Point Bend Testing
Limestone_B-72_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_B-44_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_B-48N_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_1B-72_3B-48N_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_3B-72_1B-48N_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_A-11_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_Control_NH_21-30 (no heat)	N/A	N/A	4/5/2017
TerraCotta_B-72_21-30	4/7/2017	4/24/2017	5/1/2017
TerraCotta_B-44_21-30	4/7/2017	4/24/2017	5/1/2017

Substrate and Adhesive	Bonding Start Date	Bonding End Date	Four-Point Bend Testing
TerraCotta_B-48N_21-30	4/7/2017	4/24/2017	5/1/2017
TerraCotta_1B-72_3B-48N_21-30	4/7/2017	4/24/2017	5/1/2017
TerraCotta_3B-72_1B-48N_21-30	4/7/2017	4/24/2017	5/1/2017
TerraCotta_A-11_21-30	4/7/2017	4/24/2017	5/1/2017
Limestone_Control_NH_21-30 (no heat)	N/A	N/A	4/5/2017

A. Four-Point Bend Flexure Testing

The control group specimens were fabricated and bonded using the same method as Groups A and B and were then left to set for three weeks. The specimens were placed in an oven for 24 hours at 95° F to ensure solvent evaporation prior to testing. Following setting, four-point bend flexure testing was conducted using an Instron 5569A mechanical strength analyzer. The Instron 5569A is controlled by a proprietary software entitled Bluehill Software 2 version 2.4. The Instron graphs the load (N) versus displacement (mm) for each specimen. Flexure testing is carried out at room temperature. The load is applied with a 5 kN load cell at a rate of 0.01 mm/sec until the critical load is reached. The data are recorded by the Instron 5569A and a graph is generated for each individual specimen. The graphs show the relationship between the load (or force) and the amount of deformation of the adhesive specimens prior to failure.

B. Results and Discussion

Force (Stress) vs. Displacement graphs for all of the samples in Group C are found in Appendix 3.

Limestone

Looking first at the effects of thermal cycling on only the limestone substrate, there was an overall increase in strength (8.89%) with the thermal cycling as seen in the results of the unbroken L_CT_NA vs L_CT_NH groups — cycled vs uncycled respectively. Materials often increase in strength upon heating due to loss of moisture; however, coarse-grained calcitic materials, such as marble, will often decrease in strength with heat cycling because of the thermal expansion coefficient of calcite. This is generally less true with limestones, and is demonstrated by the results. As with Group A, for all of the limestone specimens in Group C (control group) there is a relatively low standard deviation, especially as compared to the high standard deviation observed in the terracotta specimens. In general, results indicate that all of the adhesive systems performed equally to each other, but worse than the unbroken, cycled and unbroken, uncycled groups.

Looking at the individual adhesive systems, all of the adhesives in Group C exhibit lower mechanical strength than the uncycled, unbroken limestone substrate (L_CT_NH). A-11, with an average strength of 5.37 MPa, performs the worst, while both the 1:3 and 3:1 B-72:B-48N mixtures perform best, with average strengths of 6.63 MPa and 6.75 MPa. Similarly to Group A, the range in averages is very low indicating that all the adhesive systems are performing well. There is also a low standard deviation between the average strengths for each adhesive system in Group A compared to those in Group C. As seen in Table 9, the difference in mechanical strength for each system in Group A versus the corresponding adhesive system in Group C does not consistently increase or decrease suggesting that 100 cycles is not enough to degrade the performance of the adhesive systems. The similarity in performance, small standard deviation, and the variable increases and decreases between Group A and Group C suggests that for both groups the solvent has fully evaporated from the adhesives. For the limestone specimens, the solvent can evaporate relatively quickly, in both cycled and uncycled, as it is able to escape either through the joint or by moving out into the pores of the substrate.

Table 9: Average Strength of Uncycled Limestone vs. Cycled Limestone

Substrate and Adhesive	GROUP C - uncycled Average Strength (MPa)	GROUP A - cycled Average Strength (MPa)
L_B72	6.01	6.53
L_B48N	6.54	6.21
L_B44	6.45	6.57
L_A11	5.38	6.09
L_1B72_3B48N	6.63	6.42
L_3B72_1B48N	6.75	6.67
L_CT_NA (control cycled)	N/A	8.06
L_CT_NH (control uncycled)	7.34	N/A

Terracotta

For the terracotta specimens in Group C, there is an overall increase in strength with a significant standard deviation in each sample set. Like the limestone substrate, the unbroken terracotta control groups — cycled TC_CT_NA vs uncycled TC_CT_NH— exhibit an increase in strength (14.8%) with the thermal cycling due to loss of moisture. As seen in Group A, the adhesive systems in Group C perform better overall with the terracotta than with the limestone.

Looking specifically at each adhesive system, all of the adhesives, with the exception of the B-72 and the 3:1 B-72:B-48N systems, were stronger than the terracotta substrate material (TC_CT_NH). Unlike the terracotta results in Group A, which exhibit a wide range in strengths, all of the adhesive systems in Group C perform almost equally to each other — the range in strengths for Group A equals 3.01 and for Group C equals 1.53. B-44 exhibits the highest strength for both the uncycled and cycled groups, while B-72 and the 3:1 B-72:B-48 mixture exhibit the lowest strengths for both the uncycled and cycled groups. In both Groups A and C, the 1:3

B-72:B-48N mixture and B-48N perform very similarly, as do the B-72 and the 3:1 B-72:B-48 mixture, suggesting that the mixtures are controlled by the resin in greatest concentration.

Comparing Group C and Group A overall, results show a consistent increase in strength for each adhesive system with thermal cycling. These results, when compared to the limestone, suggests that the use of a barrier layer impedes solvent evaporation and lowers the performance of the adhesives. At the same time, exposure to elevated temperatures through thermal cycling accelerates solvent evaporation and thus increases the performance of the adhesive systems. In other words, 100 cycles between 30-60° C actually improves the performance of the adhesive systems — increasing the average strength from Group C to Group A — by driving off the residual solvent faster than the solvent can evaporate in the uncycled specimens due to the presence of the barrier layer.

Table 10: Average Strength of Uncycled Terracotta vs. Cycled Terracotta

Substrate and Adhesive	GROUP C - uncycled Average Strength (MPa)	GROUP A - cycled Average Strength (MPa)
TC_B72	9.11	10.08
TC_B48N	9.33	11.79
TC_B44	10.42	13.09
TC_A11	10.06	11.40
TC_1B72_3B48N	9.32	12.22
TC_3B72_1B48N	8.89	10.69
TC_NA (control cycled)	N/A	10.76
TC_NH (control uncycled)	9.17	N/A

3.4 Experimental Testing — Group B

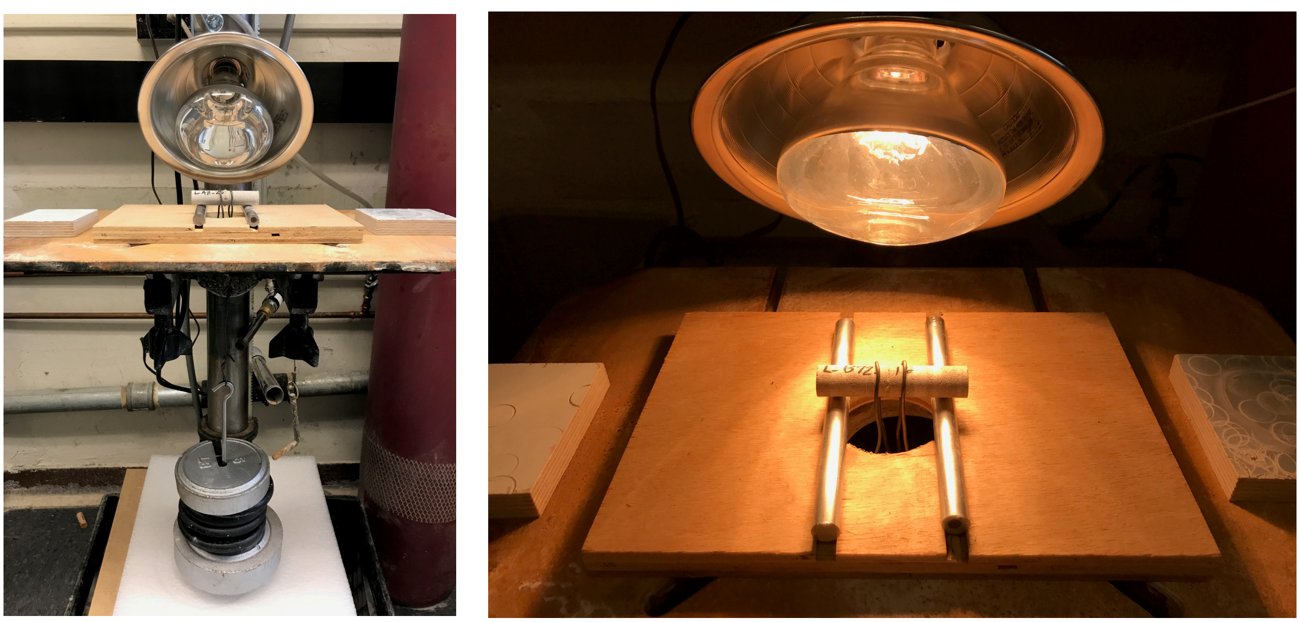
Table 11: Summary of Appendix 4 for Group B

Substrate and Adhesive	Bonding Start Date	Bonding End Date	Thermal Creep Testing Date
Limestone_B-72_11-20	2/3/2017	3/13/2017	3/29/2017
Limestone_B-48N_11-20	2/3/2017	3/13/2017	3/30/2017
Limestone_B-44_11-20	2/3/2017	3/13/2017	3/30/2017
Limestone_1B-72_3B-48N_11-20	2/3/2017	3/13/2017	3/30/2017
Limestone_3B-72_1B-48N_11-20	2/3/2017	3/13/2017	3/29/2017
Limeston_A-11_11-20	2/3/2017	3/13/2017	4/03/2017
Limestone_CT_NA_11-20 (unbroken control)	N/A	N/A	4/03/2017
TerraCotta_B-72_11-20	2/1/2017	3/13/2017	3/30/2017
TerraCotta_B-48N_11-20	2/2/2017	3/13/2017	4/03/2017
TerraCotta_B-44_11-20	2/2/2017	3/13/2017	3/30/2017
TerraCotta_1B-72_3B-48N_11-20	2/2/2017	3/13/2017	3/30/2017 (11-13) 4/03/2017 (14-20)
TerraCotta_3B-72_1B-48N_11-20	2/2/2017	3/13/2017	3/30/2017
TerraCotta_A-11_11-20	2/2/2017	3/13/2017	4/03/2017 (11-17) 4/04/2017 (18-20)
TerraCotta_Control_NA_11-20 (unbroken control)	N/A	N/A	4/04/2017

A. Stress - Creep Behavior Testing

Group B specimens were tested for thermally induced stress - creep behavior in the adhesive systems. Each specimen was placed in a steel wire apparatus such that two rings of the wire were placed on each side of the adhesive joint. The specimen was placed on two steel cylinder supports. The wire passed through a hole in the supporting work bench allowing for 20 lb. of weight to be suspended approximately 50 cm below the specimen. The A-11 terracotta samples did not fail with the 20 lb. and were therefore increased to 25 lb. in order to cause failure in the adhesives. As seen in Figure 6 below, an infrared lamp was placed 6.5 cm above the specimens at a 45° angle. The testing procedure included: adding weights to each specimen while the lamp was turned off, taking an initial temperature reading of the joint, turning on the infrared lamp and beginning the timer, monitoring temperature of the adhesive joint with a hand-held IR thermometer from a distance of approximately 83 cm and at an approximate 45° angle above the shadow cast on the specimen. Temperatures were monitored at 20 second intervals until the time of failure. The limitations of this method includes a slight variation in temperature readings as a result of using a hand-held thermometer. Additionally, this method provides a reading for the surface temperature only and does not indicate how long it takes for the heat to permeate the material. The overall configuration of the setup produced an accessory similar to the four-point bend accessory used for the Group A and Group C specimens. The design of this test may have more significance in an architectural context in which a bonded element is exposed to significant solar radiation as compared to objects indoors or stored on an archeological sites where there is limited exposure to direct sunlight.

Figure 6: Stress-Creep Behavior Testing



Stress - Creep Testing: Infrared lamp above the specimen and 20 lb. of weight suspended below.

B. Results and Discussion

Chart for all of the samples in Group B are found in Appendix 4.

Table 12: Summary of Results for Stress-Creep Testing

Substrate and Adhesive	Average Time to Failure (°C)	Average Temp at Failure (°C)	Standard Deviation for Time	Standard Deviation for Temperature
L_B72_11-20	3.50	51.04	0.73	1.99
L_B48N_11-20	5.31	61.35	0.91	5.15
L_B44_11-20	6.94	65.42	0.95	1.95
L_A11_12-20	17.86	77.50	4.14	1.74
L_3B72_1B48N_12-20	4.14	62.74	0.71	1.24
L_1B72_3B48N_12-20	5.80	62.47	0.81	3.01
TC_B72_11-20	4.66	63.11	0.81	2.15
TC_B48N_11-20	6.52	67.66	1.26	1.71
TC_B44_11-20	6.47	66.04	0.80	1.05
TC_A11_12-20 (+5 lb)	14.47	74.39	6.39	2.97
TC_3B72_1B48N_12-20	4.65	63.98	0.97	1.18
TC_1B72_3B48N_12-20	5.76	64.04	0.46	2.83

Limestone

For the limestone specimens, overall the adhesive systems exhibited longer times to failure and reached much higher surface temperatures as their respective T_gs increased.[107] The B-72 specimens, which have the lowest T_g, experienced dripping and deformation before complete failure — in one case the adhesive did not fully fail indicating that the adhesive was heating through the in the direction of the infrared radiation. Compared to B-72, the temperatures and time to failure increased for the B-48N, B-44, and significantly for the A-11. Looking at the 1:3 and 3:1 mixtures,

[107] Each specimen was timed with a stop watch from the moment the infrared lamp was turned on until the adhesive joint failed and the specimen broke in half. This is duration of time is referred to as "time to failure."

the temperatures at failure for the 1:3 and 3:1 adhesive mixtures were not significantly different from each other. However, when compared to the B-72, both mixtures exhibited longer times to failure and higher temperatures. Compared to the B-48N the time to failure and temperatures are similar, which begs the question, does B-48N control the performance of adhesive?

Terracotta

Comparing the results of the limestone specimens to the terracotta specimens, it is clear that the substrate makes a difference. Overall, the adhesives exhibited higher temperatures and longer times to failure with the terracotta. As with the limestone, there is a correspondence between increasing times to failure and surface temperatures as the adhesive T_gs increase. Additionally, the range in times to failure and temperature at failure between the B-72, B-48N, and B-44 terracotta adhesive groups is smaller than those seen in the B-72, B-48N, and B-44 limestone groups, indicating that the adhesives perform better with the terracotta substrate. The A-11 specimens, which required an additional 5 lb., continue to exhibit the highest temperatures and longest times to failure. Again, for the 1:3 and 3:1 B-72:B-48N mixtures the temperatures at failure were not significantly different from each other. When compared to B-72 and B-48N, the times to failure for both mixtures were longer than B-72 and similar to those of B-48N. However, the temperature at failure for the both the mixtures was similar to the temperatures seen in the B-72 and B-48N results. When we compare the 1:3 and 3:1 mixtures of the limestone to those of the terracotta we see that the temperatures for the limestone are slightly lower, however the time to failure is very similar between the two materials.

Looking at both substrates and their respective adhesives overall, the data revealed that while the temperatures are similar, there is a difference in the time to failure for the 1:3 and 3:1 mixture when compared to the B-72 and B-48N specimens. The increased time to failure indicates that the 1:3 and 3:1 mixtures perform better than the B-72 in both the terracotta and the limestone —

confirming the field experiments of many conservators.[108] Additionally, it is important to note that all of the specimens broke in the adhesive joint, indicating that in high thermal stress environments the adhesive is affected. These results differ from those seen in the four-point bend test results where failure generally occurred next to the adhesive joint (see Appendix 2) indicating that failure was not a result of the adhesive.

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[108] Donna Strahan and Simone Korolnik, "Archeological Conservation," *Studia Troica Monographien* 5 (2014): 521-523.; Sara A. Boy and Ida Pohoriljakova, "A Re-evaluation of Adhesives Used for Mending Ceramics at Kaman-Kalehöyük: A Final Assessment," *AAS XVIII* (2013): 83-92.

SECTION 4: CONCLUSIONS

4.1 Limestone Conclusions and Analysis

The results of the four-point bend tests reveal that the strengths of the different adhesive systems were very similar to each other for both Group A and Group C. This suggests that thermal cycling did not have much of an effect on the strength overall. However, these results are based on only 100 cycles, and potentially more cycles would cause greater divergence in the specimens. One possible conclusion for the similarities in mechanical strength between the two Groups could be the lack of residual solvent. As discussed in Section 1.4, in a polymer solution solvents act as a plasticizer, lowering the T_g and thus changing the performance of the adhesive over time as the solvent evaporates.[109] Since solvent retention can affect the mechanical properties of the dried polymer film, we would expect to see a difference in performance between Group A and Group C (as we do for the terracotta specimens) if there was any residual solvent remaining in the uncycled Group C. However, the results show no significant difference between the two groups indicating that the solvent has almost fully evaporated in the uncycled Group C and has most likely fully evaporated in the cycled Group A due to the elevated temperatures from thermal cycling.

For the stress-creep behavior tests, there was generally an increase in time and temperature to failure as the T_gs of the adhesive systems increased. This indicates that there is a relationship between the T_g and failure though the exact extent of the relationship is still not entirely clear.

One interesting observation for both the four-point bend flexure testing and the stress-creep behavior testing, was the results for the 3:1 and 1:3 mixtures of B-72:B-48N. In four-point bend, for both Group A and Group C, the two systems performed similarly, though the results were closer to the B-48N results than to the B-72. For the stress-creep behavior, the mixtures had similar temperatures at failure thought the times to failure were different — 1:3 B-72:B-48N failed in a

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[109] Eric F Hensen, "The Effects of Solvent Quality on Some Properties of Thermoplastic Amorphous Polymers Used inConservation," *Material Research Society Proceedings* Vol 352 (1996) 807-810.

shorter amount of time than the 3:1 mixture. In both cases, the times and temperatures at failure were closer to those of B-48N than of B-72. These results suggest that perhaps B-48N, even in a small amount, is controlling the performance of the system.

4.2 Terracotta Conclusions and Analysis

The results of the four-point bend tests reveal a consistent increase in strengths from the uncycled Group C to the cycled Group A. As discussed with the limestone, the consistent increase suggests that there is residual solvent still present in Group C and not in Group A. The barrier layer, applied only to the terracotta substrate, inhibited solvent evaporation for Group C by blocking the solvent from moving into the substrate and therefore allowing for evaporation through the adhesive joint only. For Group A, the exposure to higher temperatures from thermal cycling accelerated the solvent evaporation through the joint and thus increased the mechanical strength of the adhesive. In the conservation field, this means that for conservators using a barrier layer under normal circumstances, residual solvent will remain in the adhesive longer and bonded materials will therefore need to be supported for the prolonged period — whether though clamps, external armature, etc. — to account for the extended setting times. These results confirm that heating the adhesive, to an unknown extent, accelerates solvent evaporation and provides a significant initial increase in mechanical strength. Further study should examine if conservators using a barrier layer in hotter environments experience shorter setting times, and the extent to which the elevated temperatures improve the adhesive system without negatively affecting the penetration and flexibility of the adhesive. This thesis did not reach a point where thermal cycling diminished the performance of the adhesive, indicating the 100 cycles between 30-60° C is still improving the system by driving off residual solvent.

Almost all of the adhesives, with the exception of B-72, created an improved system over the unbroken terracotta substrate. This may indicate that these other adhesives would be better for terracotta than B-72 in hot environments. Especially interesting are the B-44 results which showed a significant improvement compared to the other adhesives. For the 3:1 and 1:3 mixtures of B-72:B-48N there was a greater difference in strength as compared to the limestone mixture results. However, both mixtures performed better than B-72 and in the case of the 1:3 mixture, better than the B-48N — again possibly suggesting that the B-48N, even in small amounts, is controlling the performance of the adhesive system.

For the stress-creep behavior tests, B-72 performed the worst, though it still exceeded its T_g by more than 20° C. In the case of every adhesive, the T_gs were exceeded at the surface as failure did not occur below 60° C. However, in the case of A-11 where the T_g was not exceeded, the reason for failure could be due to loss of cohesion or thermal expansion of the resin and not failure of the resin. This means that the higher temperature could have caused the adhesive to expand more with A-11 leading to mechanical failure of the joint instead of failure due to exceeding the T_g.

4.3 Conclusions and Analysis

These two tests, the thermal cycling / four-point bend and the stress-creep behavior, were designed to emulate the working environments of many field conservators today. The environmental parameters of the thermal cycling are indicative of environments where the temperature cycles with the differences of 30° C such as outdoor storage on archeological sites. The stress-creep behavior emulates environments in which the bonded element would receive direct infrared radiation such as an outdoor sculpture, monument, or architectural ornament, or an indoor object placed near a window that receives direct sunlight.

Overall the results of these two tests confirm empirical experiments carried out by conservators working in the field over the past decades. That is, all of the adhesive systems performed well at temperatures significantly higher than their reported T_gs. However, the results also suggest that further field testing should be conducted with a wider range of adhesives than has been considered in the past. Specifically, the initial result for B-44 and A-11, which performed very well with both the limestone and terracotta substrate in the four-point bend and stress-creep behavior, are a positive indication that these adhesives could be used as a conservation adhesive in hot environments. Future studies regarding the effects of temperature on acrylic thermoplastic resins should consider the issue of solvent retention and its effect on the T_g and on the mechanical properties of acrylic adhesives.

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APPENDIX 1

All of the resins analyzed in this thesis were synthesized by the Rohm and Haas Chemical Company. Therefore, the following Appendix has been included to provide the social history and context surrounding the development of synthetic polymeric materials and their immediate and ubiquitous use across a wide array of industries, including conservation.

A Brief History of Rohm and Haas

Rohm and Haas Chemical Company was founded in 1909 by Otto Haas and Otto Röhm. Röhm was a chemist who had earned a Ph.D. in 1901 after completing his dissertation entitled "On the Polymerization Products of Acrylic Acid," in which he discovered that acrylate polymers, which are the reaction products of an acid's esters, have a unique clarity, toughness, and flexibility. Eventually, Röhm would recognize the revolutionary potential of his acrylate polymers and would refine the production process to the degree necessary to support demand on the scale that was to come. However, Röhm's acrylate polymer research was put on hold when he and his business partner, Otto Haas, realized that they could make money by manufacturing bate for leather tanners instead. Within a year, their first product, Oropon, was so successful that Haas traveled to the United States to set up a second location to meet the demands of a growing American market. On September 1, 1909, Haas opened the first Rohm and Haas Company office in Philadelphia, Pennsylvania, while Röhm continued to run the factory in Darmstadt, Germany. The business grew quickly, and in 1914 Haas opened a factory in Chicago. But by 1916 the company had outgrown their Chicago home, and Haas decided to move all of production to a brand new facility in Bristol, Pennsylvania.[110]

With the onset of World War I, anti-German sentiments ran rampant throughout the United States. Haas was forced to incorporate Rohm and Haas Company to prove to the War Trade Board

[110] Seldon Hochheiser, Rohm and Haas: History of a Chemical Company (Philadelphia: University of Pennsylvania Press, 1986), 3-36.

that Rohm and Haas, Bristol, and Rohm and Haas, Darmstadt were separate and had been operating independently. In 1920, Röhm formally resigned his share of the company to Haas. However, the two companies continued to exchange information and research on product development but with the understanding that they were independent entities.[111] This allowed Haas to benefit from Röhm's renewed interest in acrylic chemistry and the subsequent research that came out of the Darmstadt factory. Though Röhm had initially begun his research during his doctoral thesis, he did not return to the subject of acrylic chemistry for over a decade. In 1912, Röhm resumed his work on acrylic acid, obtaining a patent on acrylic ester polymers as rubber substitutes and a second patent in 1915 on polyacrylates as a replacement for drying oils and varnishes.[112] However, it was not until the 1920s that Röhm figured out how to synthesize acrylic esters from ethylene cyanohydrin on a large-scale, and by 1926 he was close to developing an industrially practical synthesis for acrylic acid. Two years later, in 1928, Röhm introduced the first commercial acrylic product to the German market, Luglas, a methyl acrylate interlayer for automotive safety glass.[113]

During the same time period, Haas set up an affiliated company, under his direction and located in the same building, called The Resinous Products and Chemicals Company. This company specialized in the development of synthetic resins formed by the condensation of a phenol derivative and formaldehyde in the presence of small quantities of rosin.[114] Throughout the 1930s Resinous Products introduced a number of new products for coating manufacture. Amber 801, introduced in 1931, was a rosin-modified maleic acid resin used primarily in nitrocellulose lacquers and the only product to achieve substantial commercial success by the mid-1930s. While Resinous Products pursued their own line of acrylic resin research, Haas and Röhm signed an agreement in 1931 in

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[111] ibid 3-36.

[112] Synthetic resins: Amberol, Duraplex, Amberlac, Paraplex, Uformite, Acryloid, Aquaplex, Oilsolate; plywood adhesives, special products (Philadelphia: Resinous Products & Chemical Co, 1941), 53.

[113] Hochheiser 36.

[114] ibid 41-43.

which Rohm and Haas, Bristol agreed to subsidize Darmstadt's acrylic research in exchange for the North American rights to its results.[115] Within the year, Haas had set up an acrylic lab in Pennsylvania and released the American version of Luglas safety glass known as Plexigum.[116] The early acrylic safety glass layers had two major problems: it performed poorly at low temperatures and its soft, rubbery consistency made it difficult to cut into various shapes and sizes. Röhm continued to try and improve the properties of Luglas; by 1931 he had created polymethyl methacrylate, the polymer of the methyl ester of methacrylic acid. Unlike the softer acrylates, polymethyl methacrylate softened around 110°C and could be easily worked with standard tools.[117] Rohm and Haas Company released the US version of the polymethyl methacrylate sheet material, known as Plexiglas, in 1936, followed by their polymethyl methacrylate molding powder, Crystalite. [118] In 1934 Resinous Products introduced their own acrylic plastic, which they named Acryloid. The earliest trademark for Acryloid was filed by Resinous Products and Rohm and Haas Company on December 12, 1934, claiming that Acryloid was a "synthetic resin capable of forming a water white transparent film for use as a protective layer or bonding agent on fabrics, metals, and other surfaces."[119]

By the mid-1930s, Rohm and Haas Co., Darmstadt was producing Luglas; Rohm and Haas Co., Bristol had Plexigum, and Resinous Products had Acryloid. However, they were not the only producers of acrylic resins; moreover, it is important to note that theirs were not the first acrylic resins used in conservation. Lucite 44 and 45, made by E.I. du Pont Nemours & Co., Inc., were released in 1937 as both an acrylic sheet and an acrylic molding powder. Lucite acrylics were

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115 ibid 56.

116 ibid 36.

117 ibid 56-57.

118 John Harry Du Bois, Plastics, a simplified presentation of the manufacture and use of the important plastics materials and products with tables of their properties and the basic design information required by engineers and designers (Chicago: American Technical Society, 1941), 85.

119 The Resinous Products & Chemical Company, "United States Trademark: 71359240 - ACRYLOID," December 12, 2934.

possibly the earliest acrylics used by conservators, primarily as picture varnishes. They were a popular choice among conservators until an early study in 1952 entitled "Plastics Aid in Conservation of Old Paintings" by Alfred Werner. He found that the poly(buytl methacrylate) (PBMA) polymer in the Lucite acrylics showed a tendency to cross-link under visible light. This tendency negated its reversibility in the long term, though it was still reversible in the short term as it was soluble in non-polar solvents.[120] By the 1950s long-term reversibility was an important tenet of conservation practice, and so conservators continued searching for more stable polymers.

Darmstadt and Bristol scientists continued to experiment with ways to overcome the technical shortcomings of the acrylate glass, while much of the laboratory work shifted instead to methacrylate as it was seen to have a greater commercial potential. As a thermoplastic, the poly(methyl methacrylate) sheets could be shaped when heated above 110 °C. They were then stretched over a form and hardened by cooling. In the United States, the first commercial markets to adopt the new Plexiglas material included spectacles, instrument covers, dentures, display cases, and lighting fixtures; though, the military aircraft industry during WWII was the primary consumer of Plexiglas.[121]

In an effort to improve the properties of Plexiglas, the acrylics research team at Resinous Products provided the Acryloids as a copolymer for Plexiglas. According to *The Resinous Reporter* from March 1940, Acryloids were first used "in fields where no other resin would serve, namely, in the manufacture of the glass-like sheet material, Plexiglas."[122] As with Plexiglas, the Acryloids were also quickly adopted in a variety of industries, such as the manufacture of dentures, due to their chemical resistance, permanence, and mechanical strength.[123] In the coatings field "the use of these

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[120] Du Bois 85-87.; A.E.A. Werner 363-366.

[121] Hochheiser 55-68.

[122] "Acryloids," The Resinous Reporter Vol I No. 1 (1940).

[123] "Acrylic Dentures," The Resinous Reporter Vol II No. I (1941).

resins is still in its infancy, but holds every promise of developing."[124] "While the Acryloids are still in the 'luxury' class as far as resins go, they frequently will do a job which nothing else will do."[125] Perhaps most relevant to this thesis, was the claim that Acryloids were "resistant to high baking temperatures, and extremely durable, colorless, protective finishes for metals, in the architecture, aircraft, and other industries." This contradicts the claim among many conservators and conservation scientists that these resins were never intended for outdoor use, because, in fact, in 1940 the company stated that these resins could be used outdoors and in high temperatures.

By 1947, Resinous Products had expanded their Acryloid trademark to a "synthetic resinous material(s) in the form of granules, powders, and solutions for the use in printing inks, coatings, films, and adhesives."[126] According to the document, Acryloid had "been continuously used and applied to said goods in applicant's business since September 14, 1934."[127] The trademark describes the use of Acryloids as "solutions of synthetic organic thermoplastic resins characterized by their water-white color, high degree of transparency, and resistance to discoloration, said solutions being in the nature of lacquers, suitable for use in pigmented or un-pigmented form as protective coatings, in Class 16."[128] This second trademark suggests that between 1940 and 1947, Rohm and Haas recognized the versatility of the Acryloids, and had already begun to move beyond the initial printing inks and dentures.

World War II again brought legal complications for Rohm and Haas in the United States. In 1942, Rohm and Haas Company, Otto Haas, and three other major chemical companies and their executives were indicted on criminal charges of conspiring to control the marketing, production, and price of acrylic products in violation of the Sherman Antitrust Act of 1890. Haas argued that he had

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124 "Acryloids" (1940).

125 "Acryloids" (1940).

126 The Resinous Products & Chemical Company, "United States Trademark: 71507846 - ACRYLOID" August 22, 1946.

127 ibid.

128 ibid.

only signed agreements with Darmstadt and I.G. Farben (the largest chemical company in Germany at the time) in order to acquire the rights to certain patents owned by German companies. Interestingly, the agreement for the 1920s between Rohm and Haas, Bristol and Rohm and Haas, Darmstadt contained specific terms that restricted Haas to marketing acrylic products in the United States and Canada while giving Darmstadt exclusive European rights to all acrylic patents that came out of the Bristol lab. In addition, Haas' agreements with I.G. Farben restricted the industries to which he could sell acrylics — safety glass, adhesives, and glass substitutes — i.e., industries in which conservators would have been involved, while I.G. Farben reserved the right to sell acrylics for synthetic rubber, photographic materials, and pharmaceuticals.[129] Though the antitrust case was eventually dropped, Haas continued to push his luck by attempting to purchase the Darmstadt company twice in the following years. The often cited, but never fully explained, name debacle of Acryloid vs. Paraloid occurred during Haas' final purchase attempt in 1951. Denied by the Justice Department for a second time, the two companies instead signed an agreement on their jointly used trademarks. The 1951 document specifies that the Philadelphia company could not use the names and prefixes Oropon, Plexi-, and Acry- in certain markets including Europe.[130] Thus, on February 26, 1952 the Paraloid name was introduced in the European market and was trademarked as "acrylic ester for inks, coatings, etc; synthetic resin and liquid solution for plastic compounds," while the Acryloid name continued to be used in the United States and Canada.[131] At the October 31, 2007 Rohm and Haas Conservation Webcast, Rohm and Haas announced the retirement of the trademark term Acryloid, stating that going forward, they would only use the term Paraloid.[132]

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129 Hochheiser 75-77.

130 Hochheiser 110.

131 Hochheiser 218.

132 Personal email communication with Stephen Koob (April 9, 2017).

APPENDIX 2

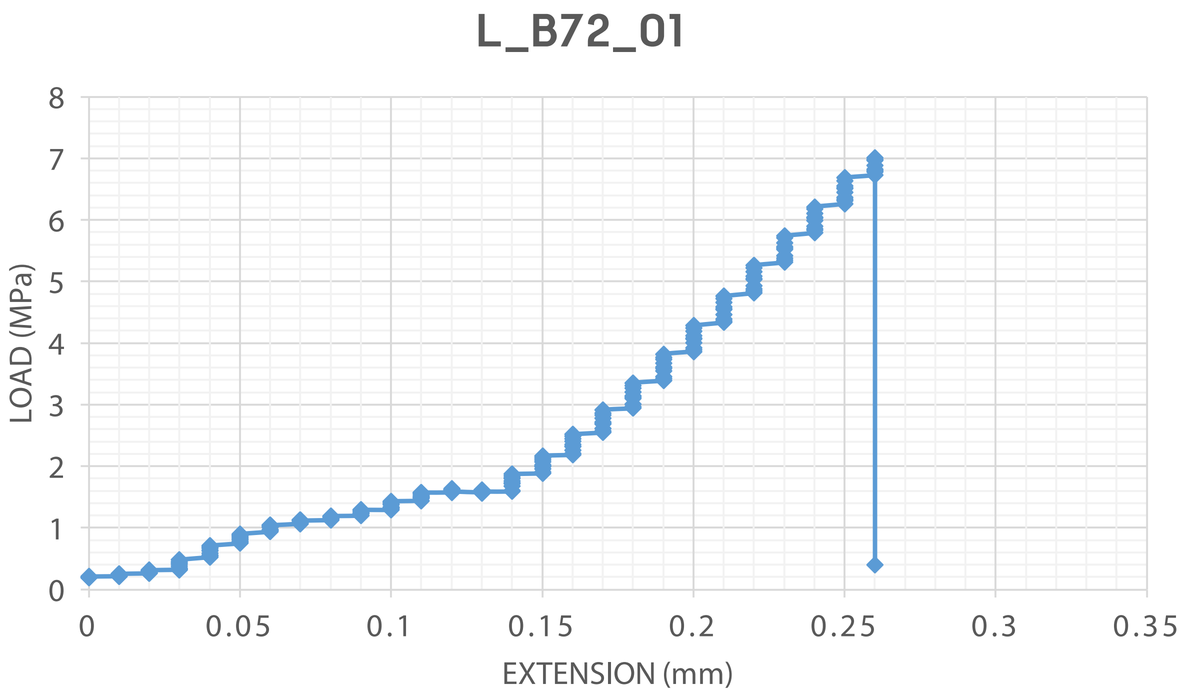
LIMESTONE – GROUP A

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
L_B72_01	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-437.67	Broke far from joint	7.18			961.30
L_B72_02	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-318.72		5.23			696.44
L_B72_03	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-482.78	Broke next to joint	7.92			1003.83
L_B72_04	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-360.10		5.91			1278.68
L_B72_05	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-441.10	Broke next to joint	7.23			1340.38
L_B72_06	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-394.13	Broke far from joint	6.46			1201.81
L_B72_07	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-419.82	Broke next to joint	6.89			1129.55
L_B72_08	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-315.68		5.18			1054.46
L_B72_09	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-418.06	Broke next to joint	6.86			978.43
L_B72_10	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-395.70		6.49			1230.27
											6.53	0.88	
L_B48N_01	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-391.97		6.43			626.76
L_B48N_02	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-337.15		5.53			759.13
L_B48N_03	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-346.76		5.69			792.22
L_B48N_04	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-383.73		6.29			884.95
L_B48N_05	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-411.00		6.74			1143.14
L_B48N_06	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-387.17		6.35			910.44
L_B48N_07	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-403.94		6.62			897.24
L_B48N_08	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-400.31		6.57			878.89

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
L_B48N_09	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-404.82		6.64			866.44
L_B48N_10	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-321.95		5.28			961.22
											6.21	0.52	
L_B44_01	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-386.58	Broke next to joint	6.34			1283.54
L_B44_02	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-356.57	No visible failure	5.85			814.59
L_B44_03	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-443.55		7.27			1243.24
L_B44_04	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-417.57		6.85			1081.25
L_B44_05	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-422.18		6.92			1041.56
L_B44_06	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-371.87		6.10			1042.74
L_B44_07	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-395.99		6.49			1090.37
L_B44_08	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-273.03	Broke next to joint	4.48			797.14
L_B44_09	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-437.28		7.17			1018.51
L_B44_10	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-502.00		8.23			1260.25
											6.57	1.00	
L_A11_01	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-403.35	No visible failure	6.61			1034.25
L_A11_02	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-379.91	No visible failure	6.23			1133.54
L_A11_03	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-317.64	No visible failure	5.21			1221.33
L_A11_04	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-378.44	No visible failure	6.21			995.17
L_A11_05	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-300.08	No visible failure	4.92			971.88
L_A11_06	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-318.03	No visible failure	5.22			848.72

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
L_A11_07	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-478.86	No visible failure	7.85			1054.98
L_A11_08	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-290.28	No visible failure	4.76			1281.11
L_A11_09	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-425.61	No visible failure	6.98			1351.08
L_A11_10	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-421.20	No visible failure	6.91			1331.95
											6.09	1.03	
L_1B72_3B48N_01	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-417.67	Broke far from joint	6.85			983.22
L_1B72_3B48N_02	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-339.70	Broke next to joint	5.57			896.38
L_1B72_3B48N_03	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-310.67		5.09			1286.28
L_1B72_3B48N_04	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-444.24	Broke far from joint	7.29			1120.18
L_1B72_3B48N_05	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-421.39	Broke far from joint	6.91			1314.51
L_1B72_3B48N_06	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-370.30		6.07			1080.43
L_1B72_3B48N_07	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-395.99		6.49			920.52
L_1B72_3B48N_08	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-416.00	Broke next to joint	6.82			1569.26
L_1B72_3B48N_09	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-408.35	Broke far from joint	6.70			826.34
L_1B72_3B48N_10	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-390.99	Broke next to joint	6.41			1229.99
											6.42	0.67	
L_3B72_1B48N_01	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-378.05	Broke next to joint	6.20			870.75
L_3B72_1B48N_02	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-429.43	Did not break fully	7.04			977.36
L_3B72_1B48N_03	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-392.36	Broke next to joint	6.43			1106.21
L_3B72_1B48N_04	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-354.31	Broke next to joint	5.81			867.00

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
L_3B72_1B48N_05	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-416.10	Broke next to joint	6.82			1450.04
L_3B72_1B48N_06	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-408.35	No visible failure	6.70			1381.98
L_3B72_1B48N_07	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-460.32	Broke far from joint	7.55			1717.45
L_3B72_1B48N_08	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-393.34	Broke next to joint	6.45			824.54
L_3B72_1B48N_09	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-369.61		6.06			1212.20
L_3B72_1B48N_10	2/3/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-465.33	Broke far from joint	7.63			1402.43
											6.67	0.60	
L_CT_NA_01	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-459.05		7.53			970.88
L_CT_NA_02	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-478.37		7.85			948.94
L_CT_NA_03	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-486.02		7.97			1208.82
L_CT_NA_04	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-492.59		8.08			1040.77
L_CT_NA_05	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-514.65		8.44			1147.15
L_CT_NA_06	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-497.00		8.15			973.62
L_CT_NA_07	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-518.09		8.50			1041.66
L_CT_NA_08	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-488.37		8.01			872.79
L_CT_NA_09	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-484.15		7.94			929.92
L_CT_NA_10	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-495.73		8.13			1073.70
											8.06	0.28	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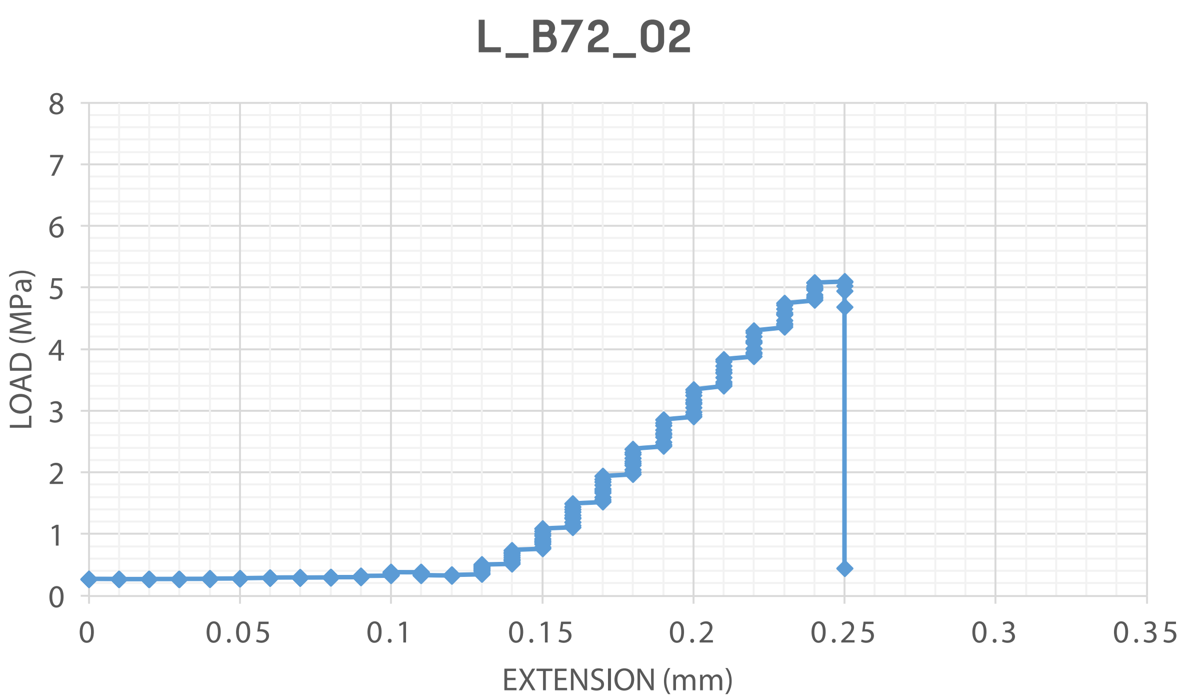
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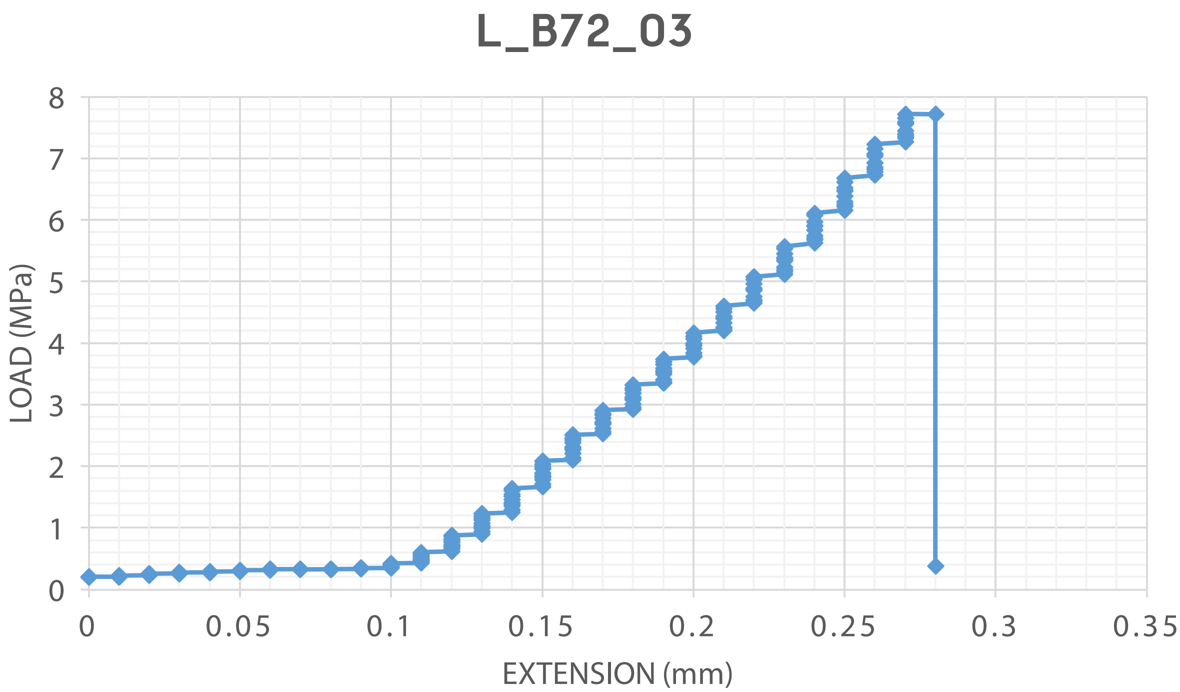
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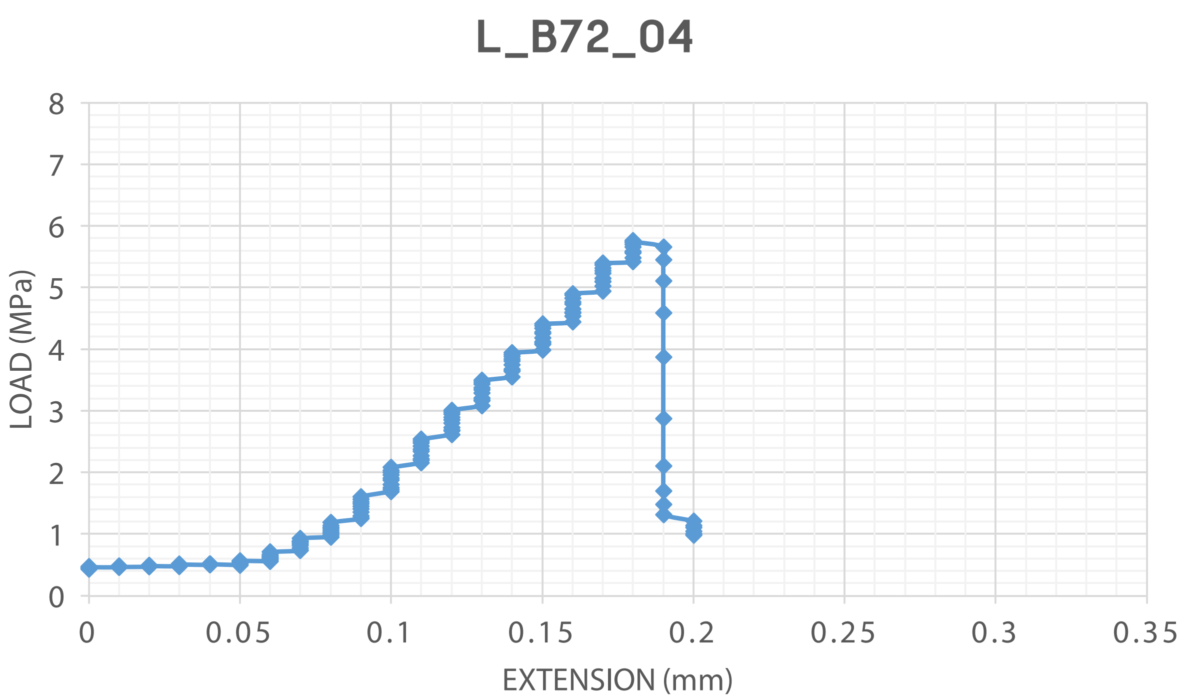
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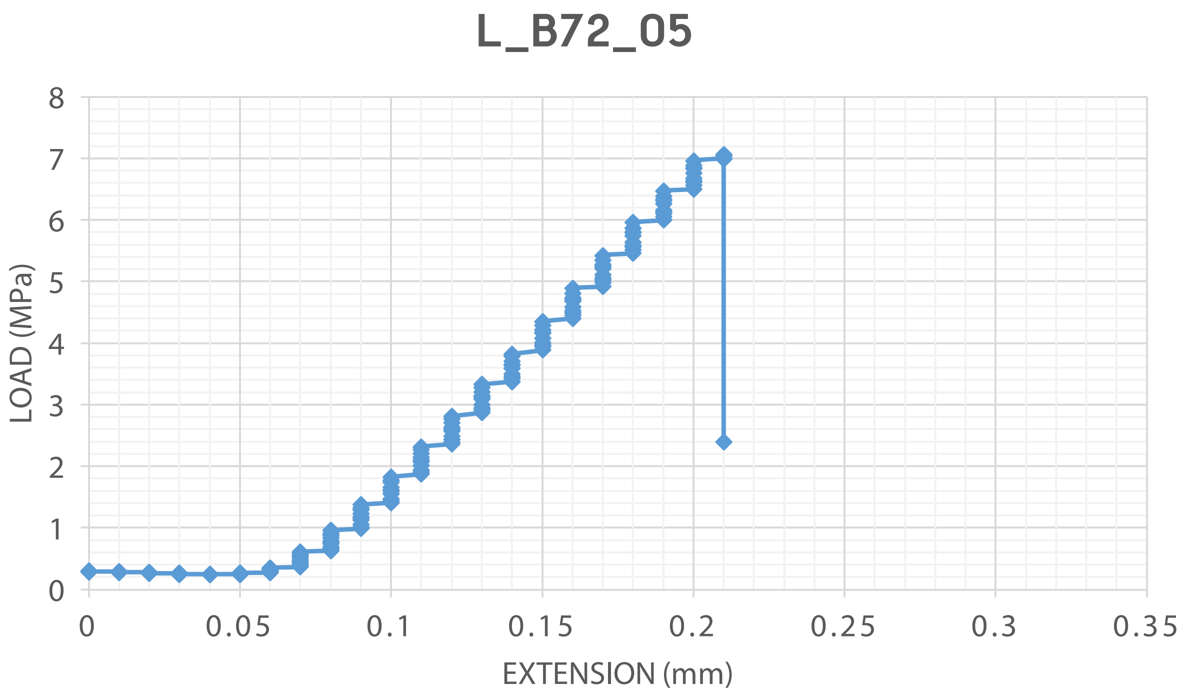
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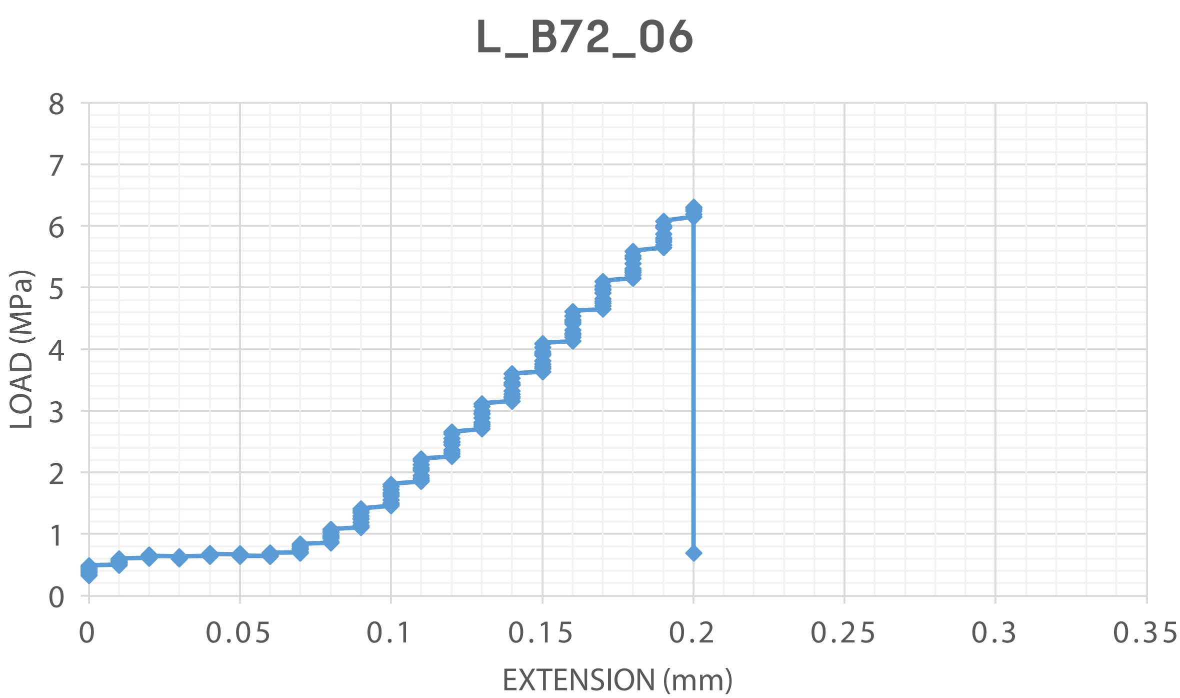
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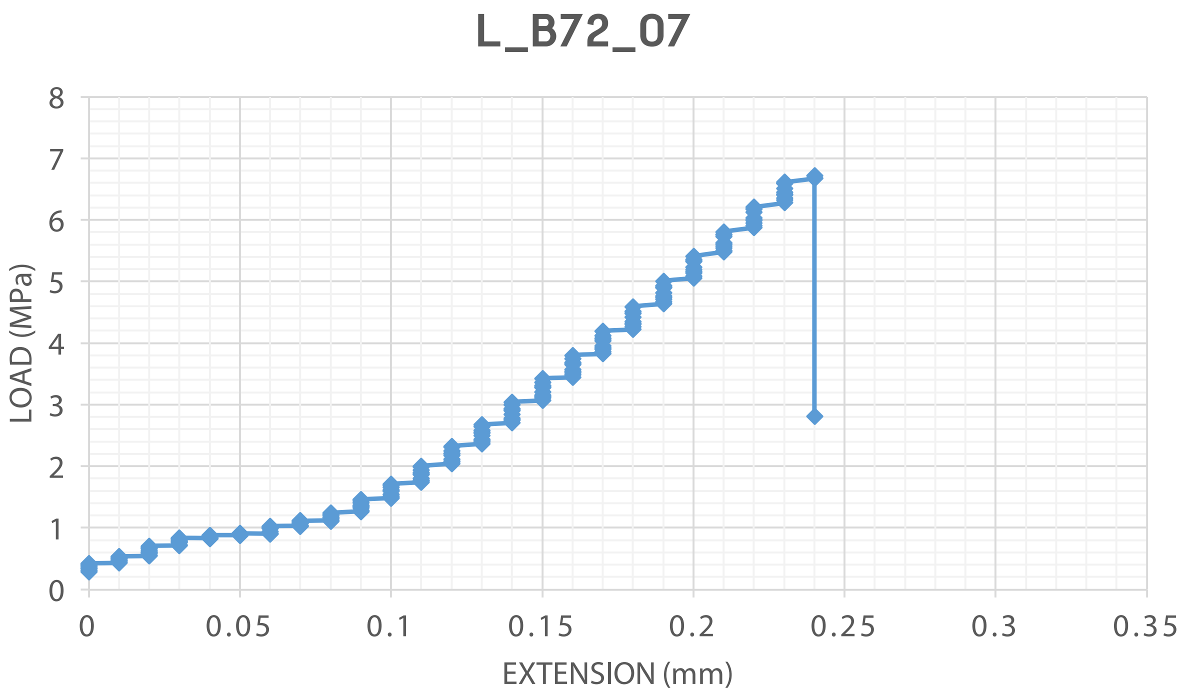
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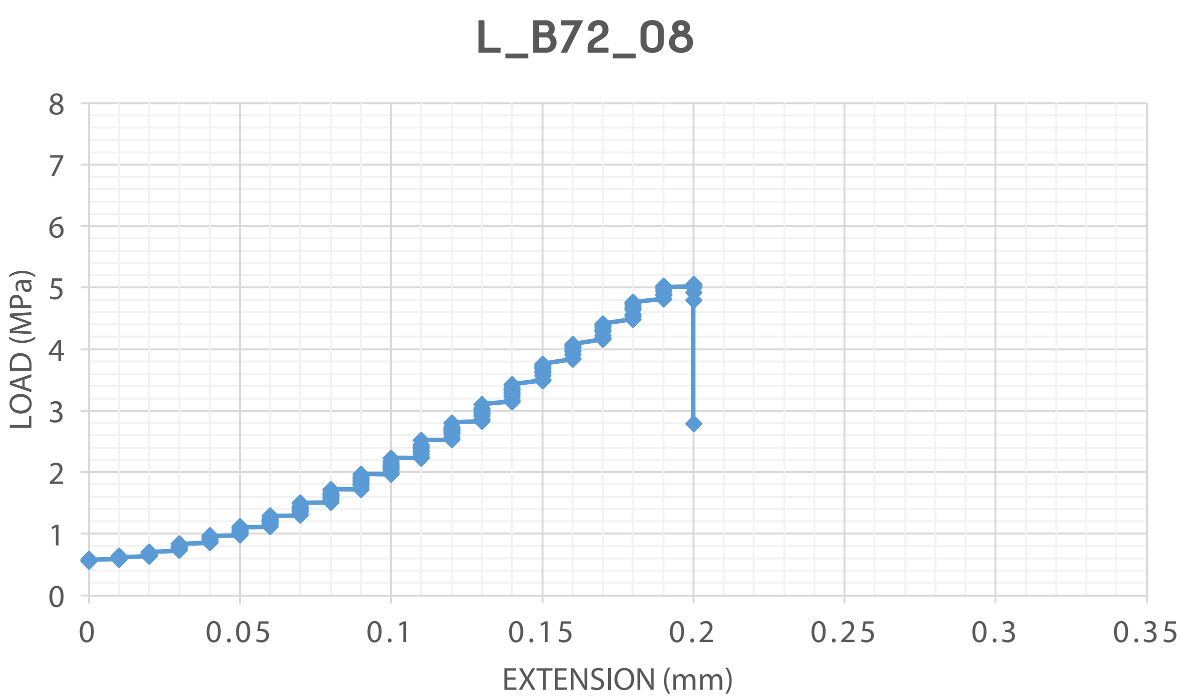
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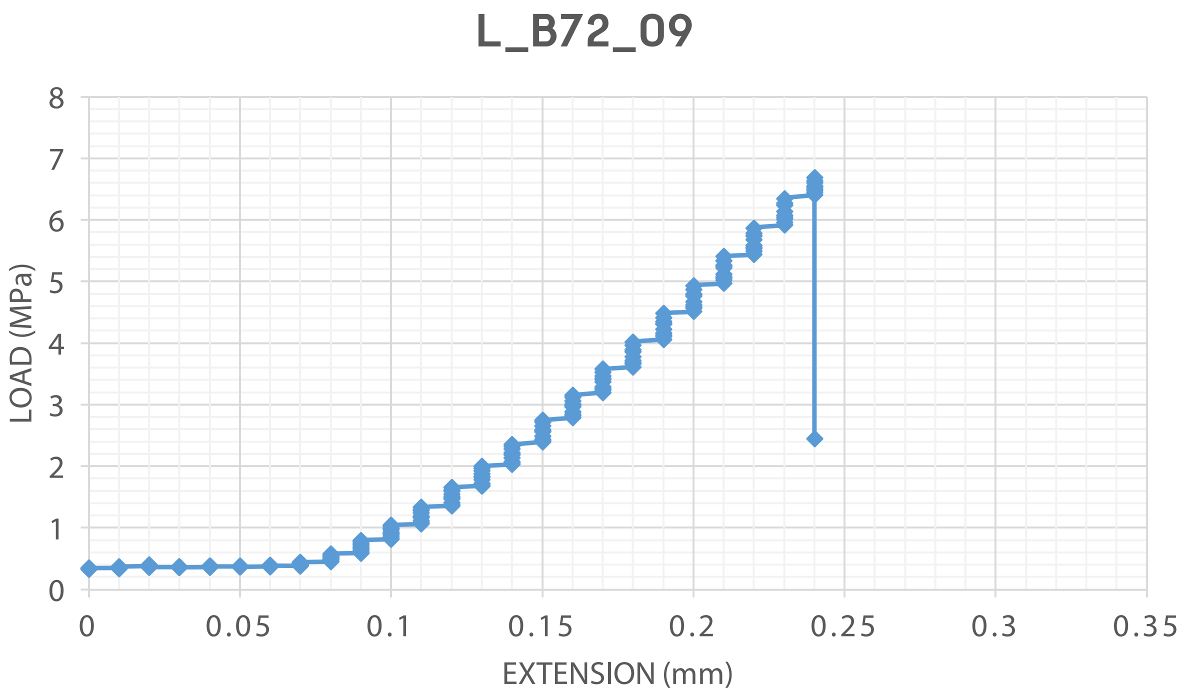
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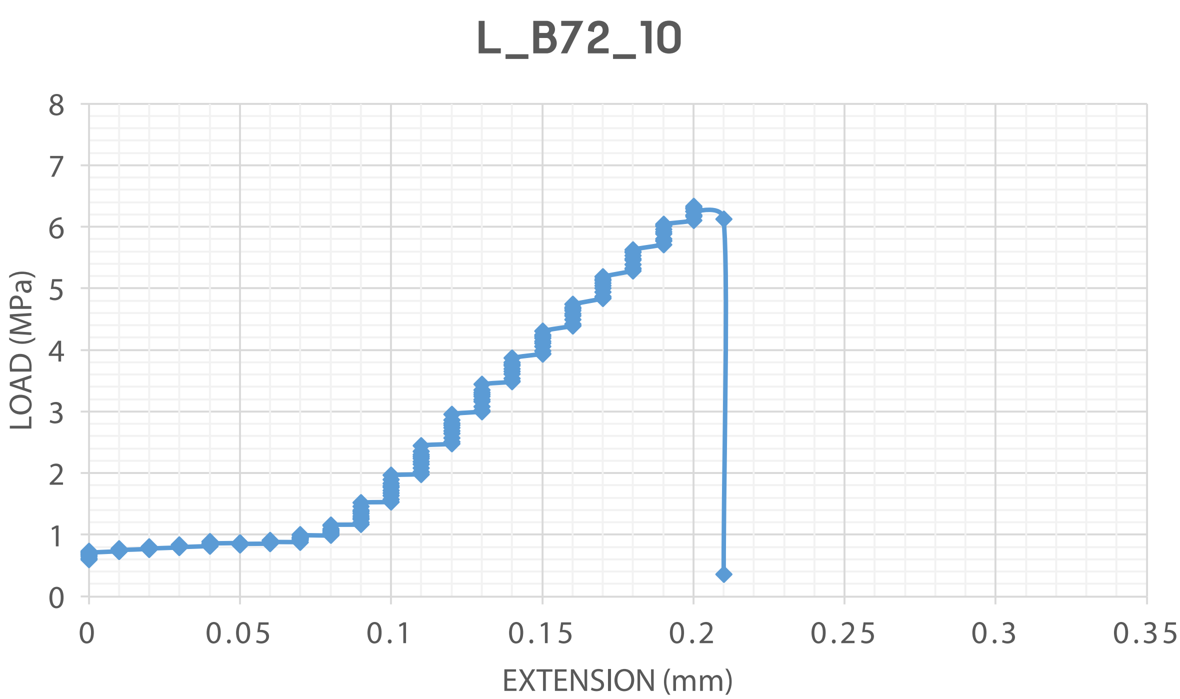
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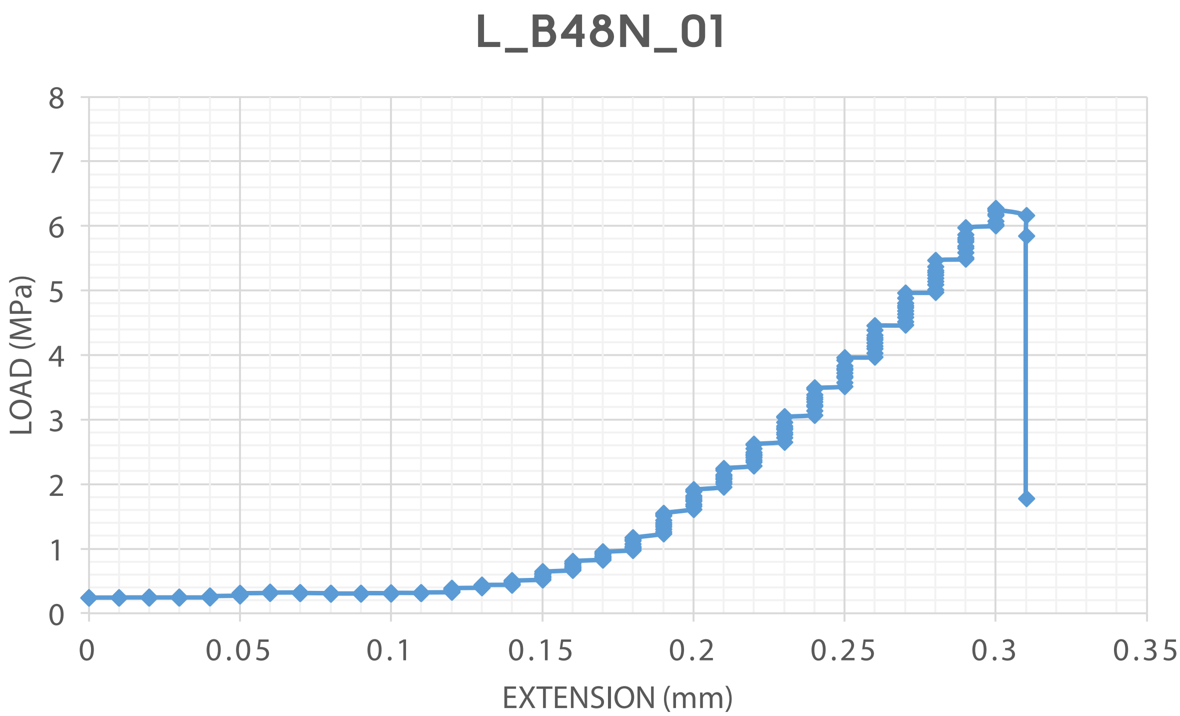
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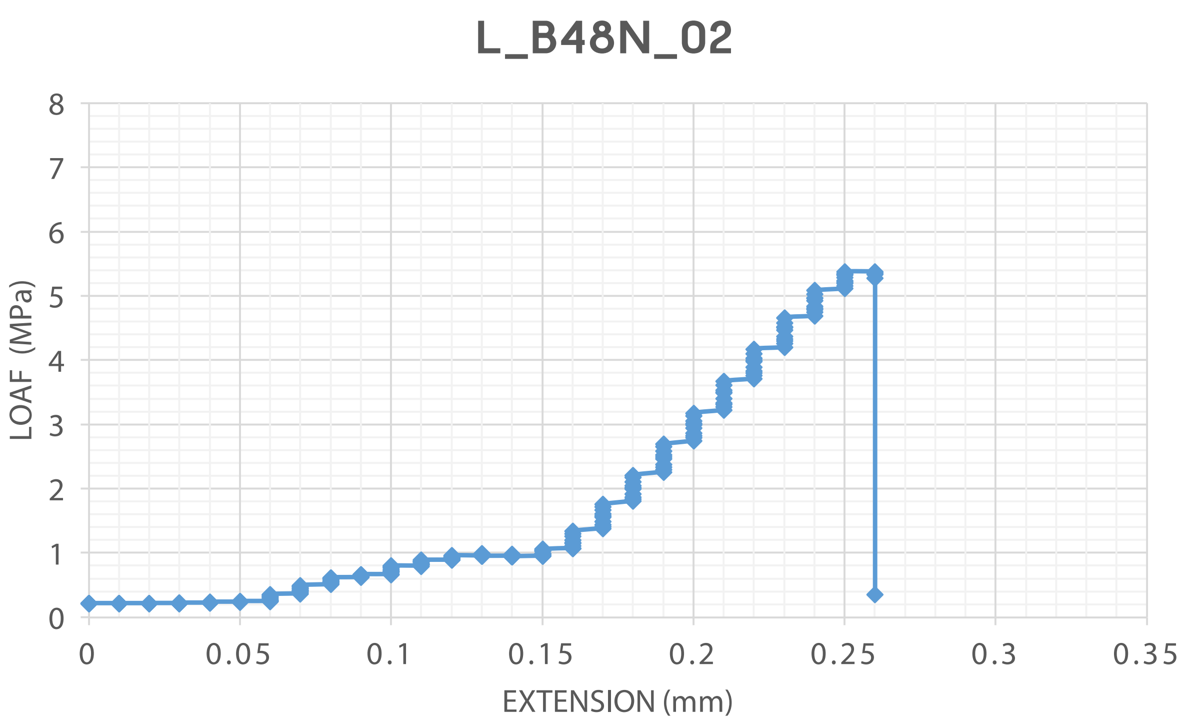
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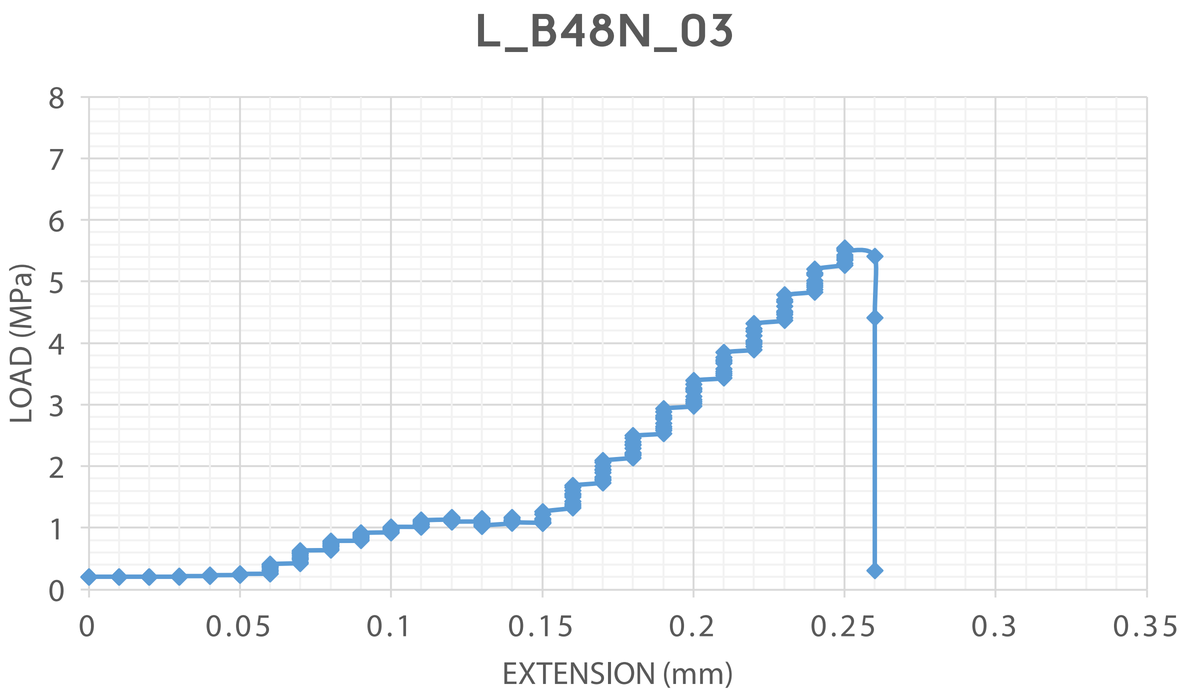
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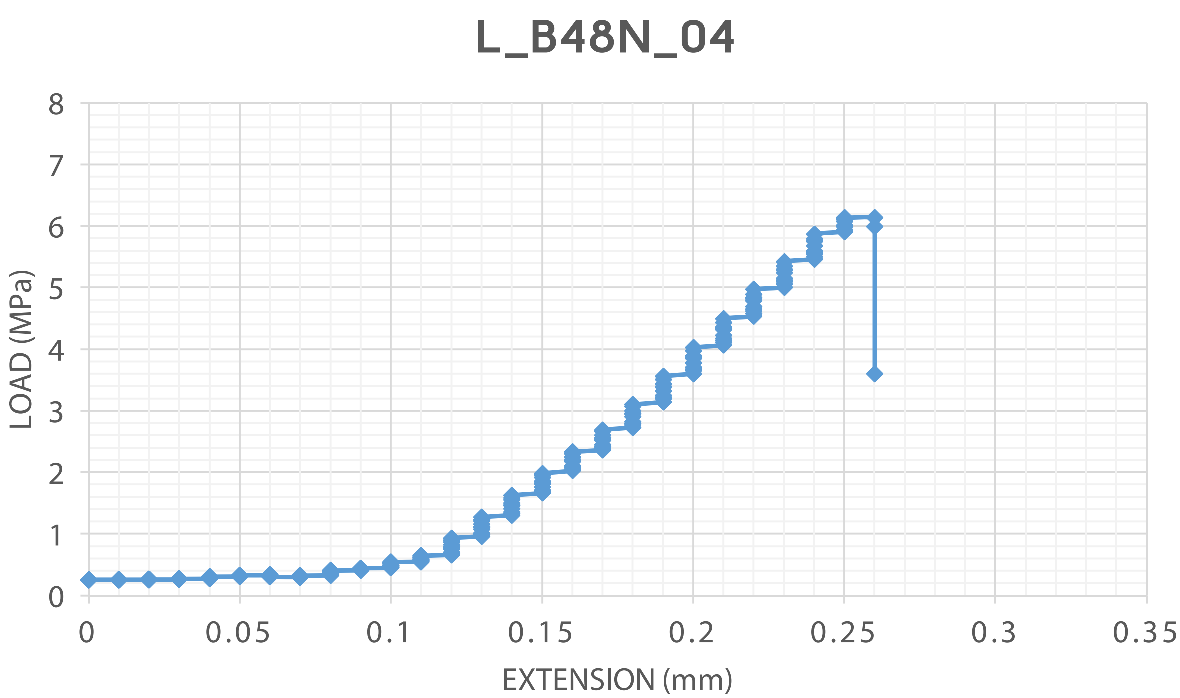
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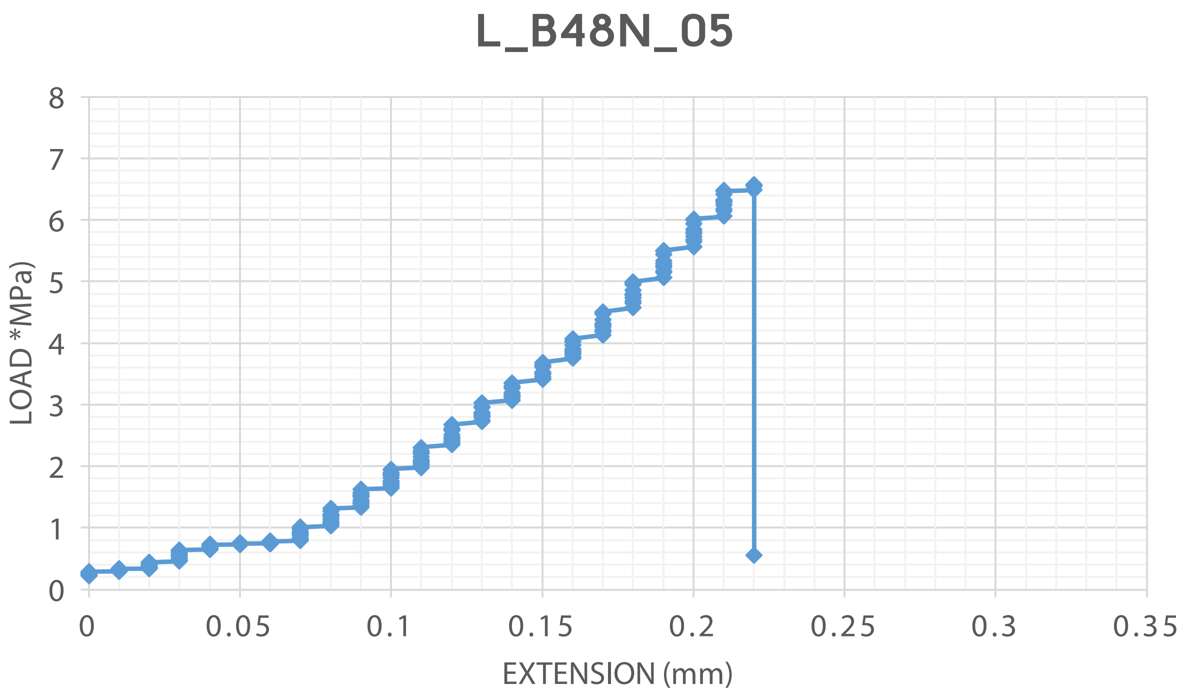
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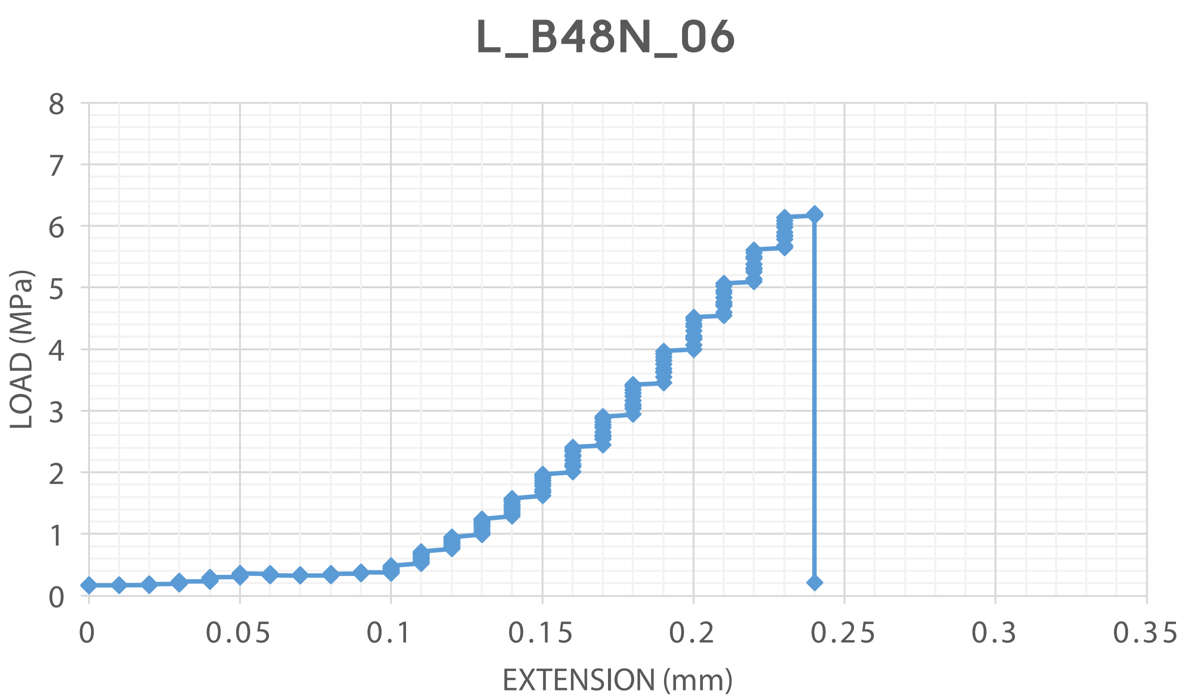
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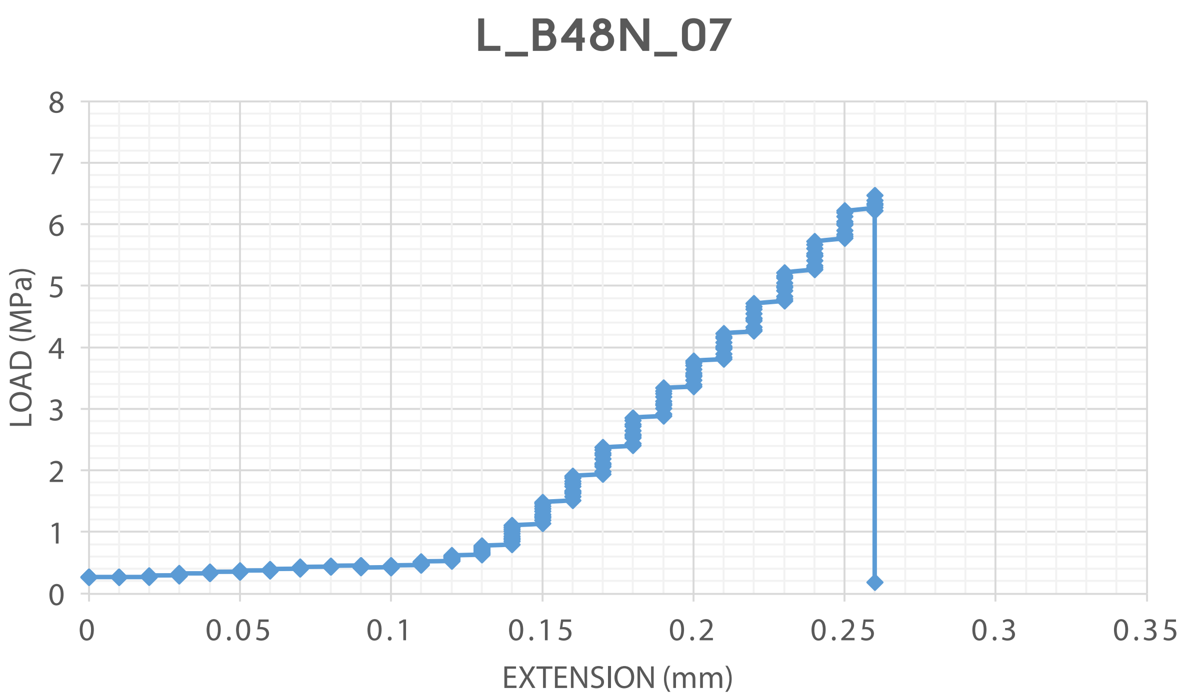
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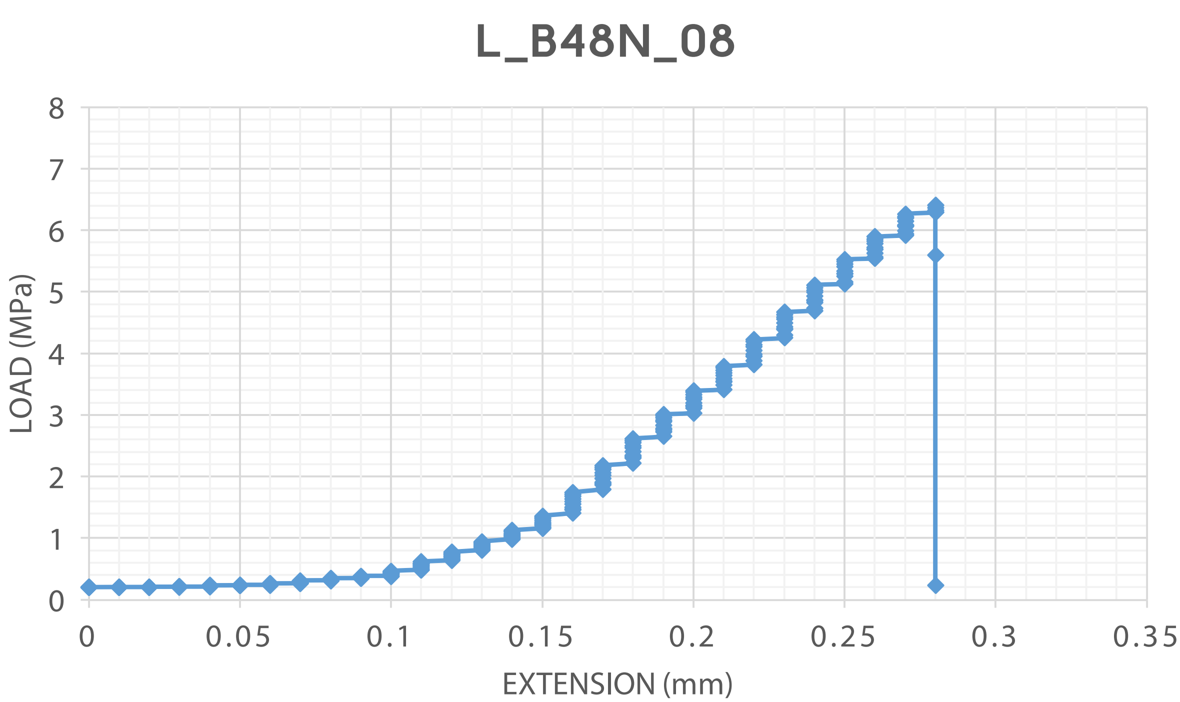
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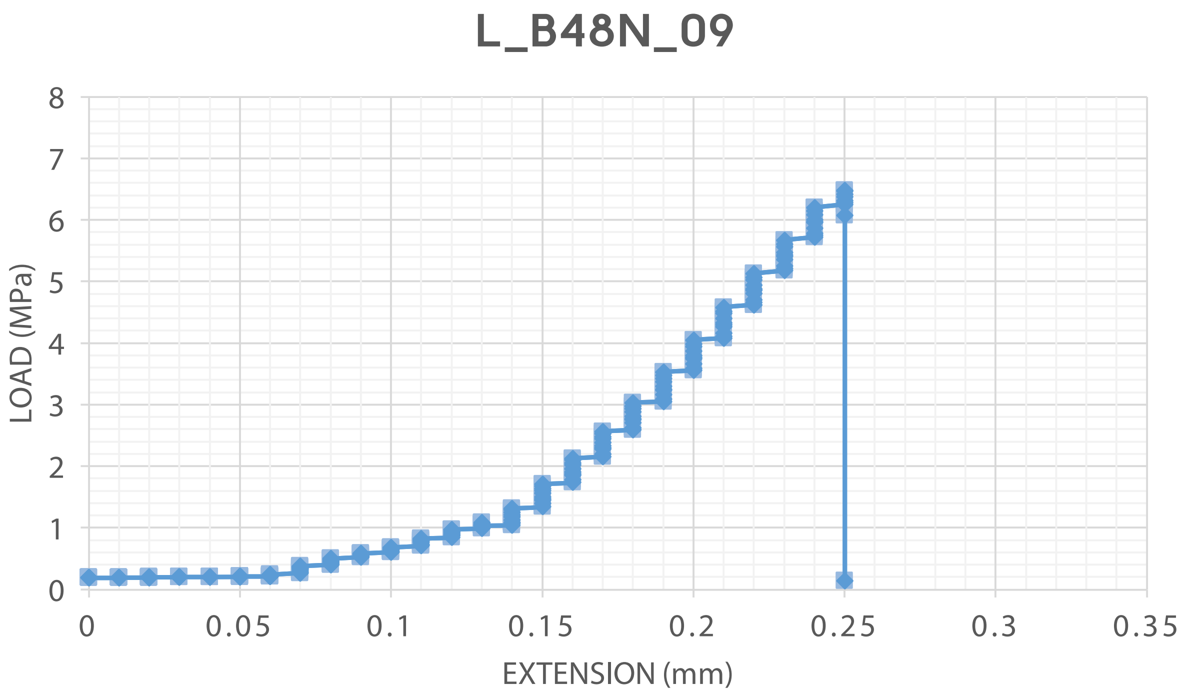
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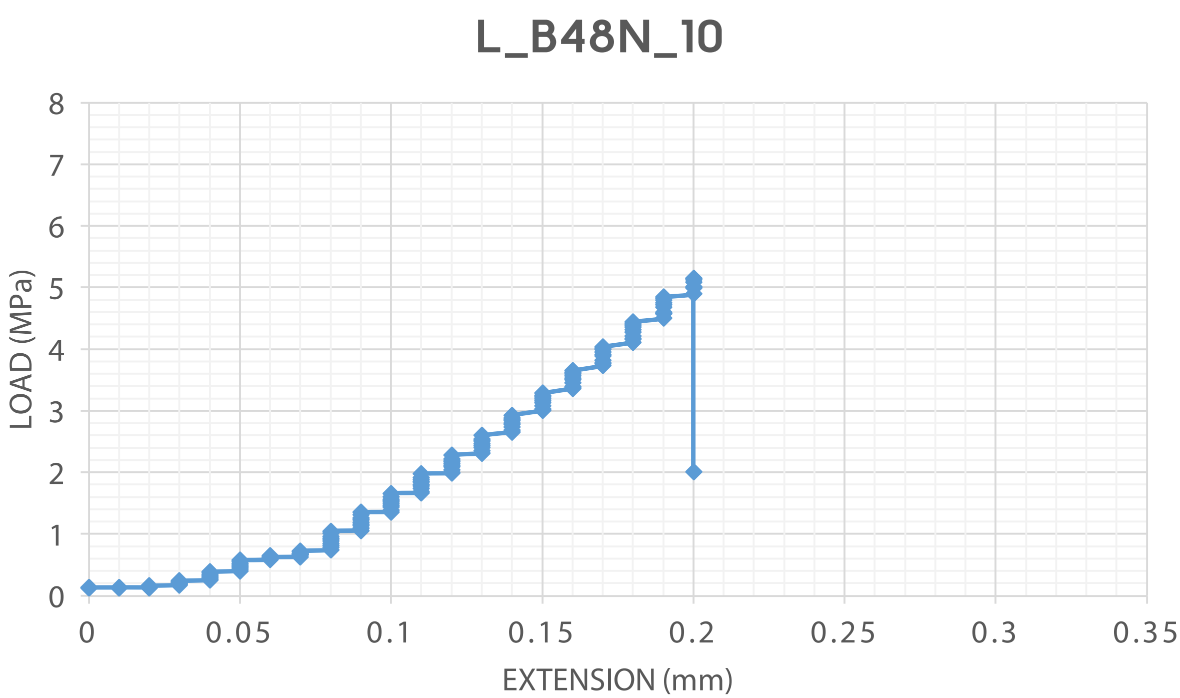
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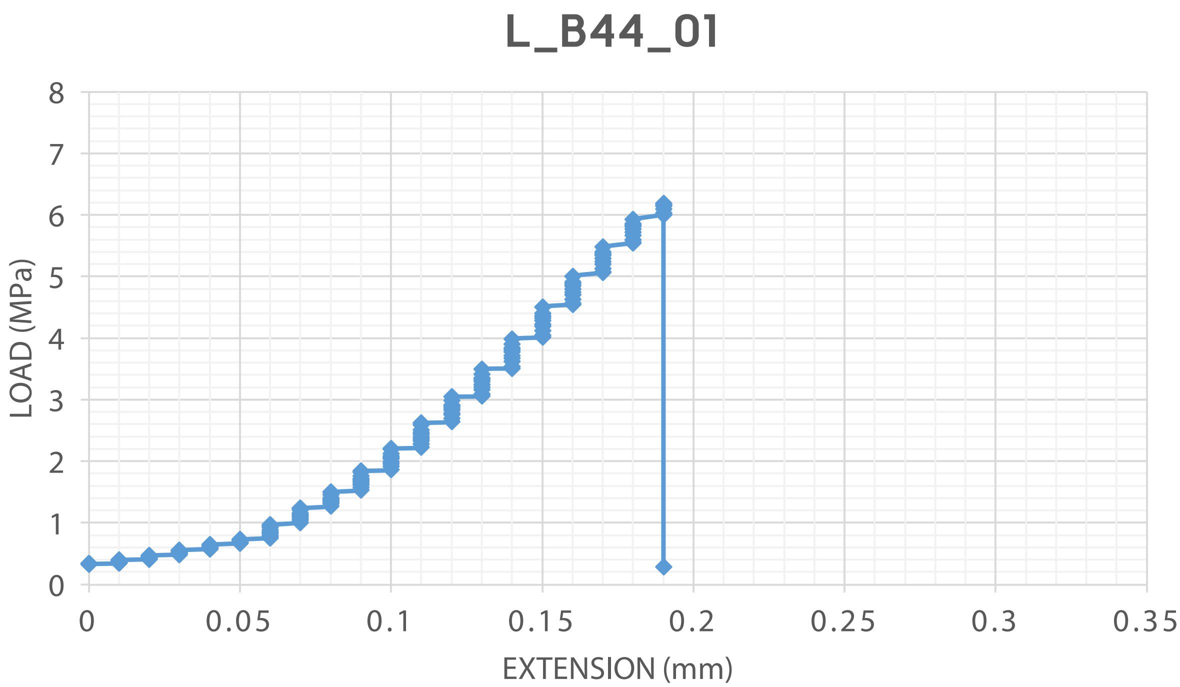

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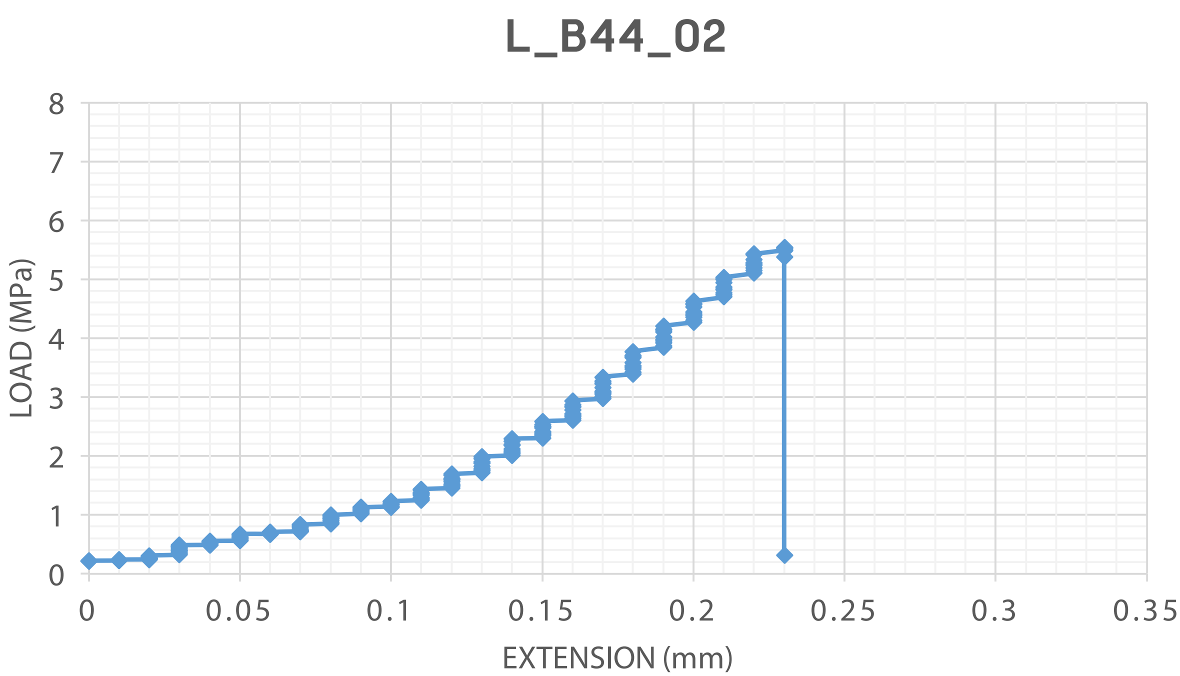

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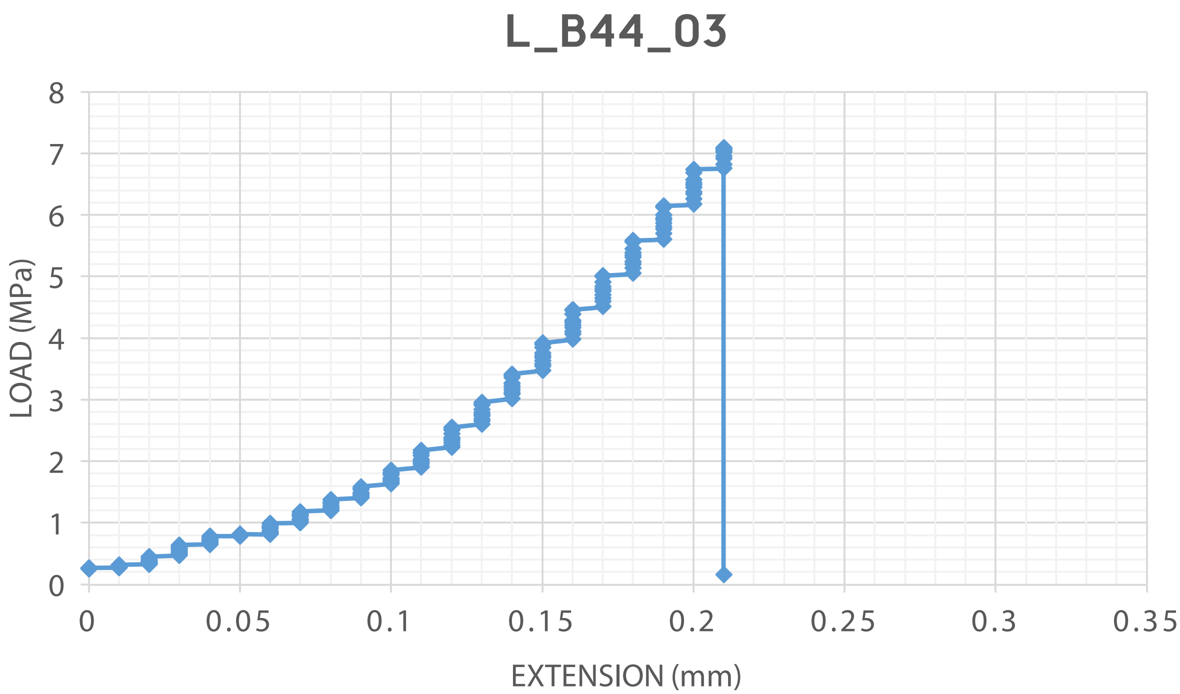
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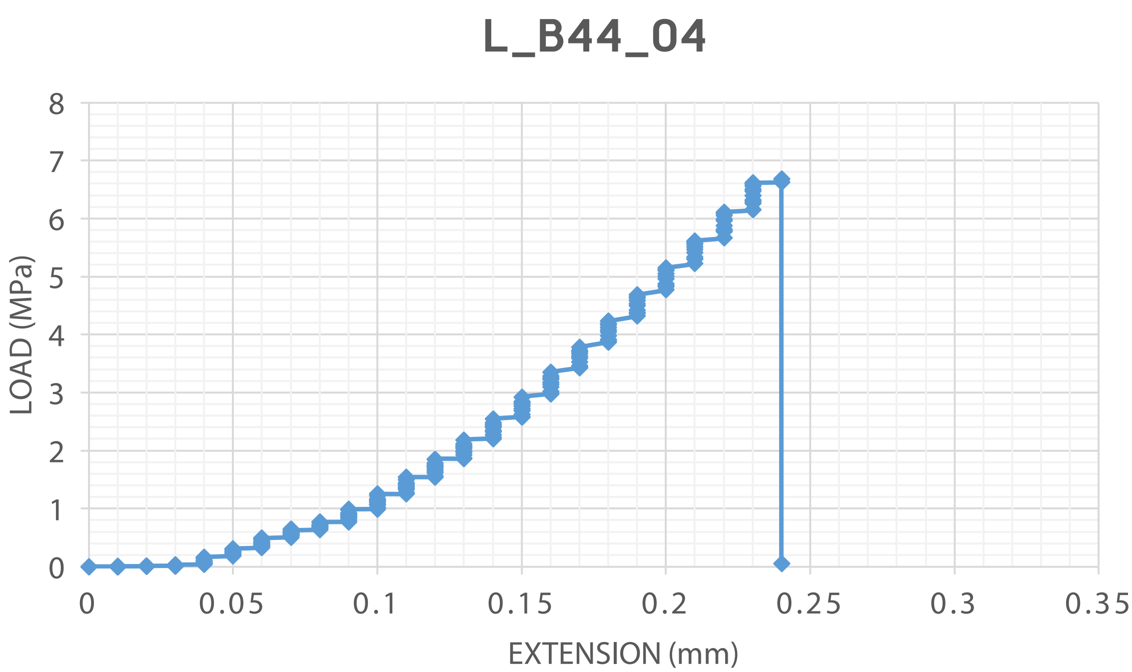
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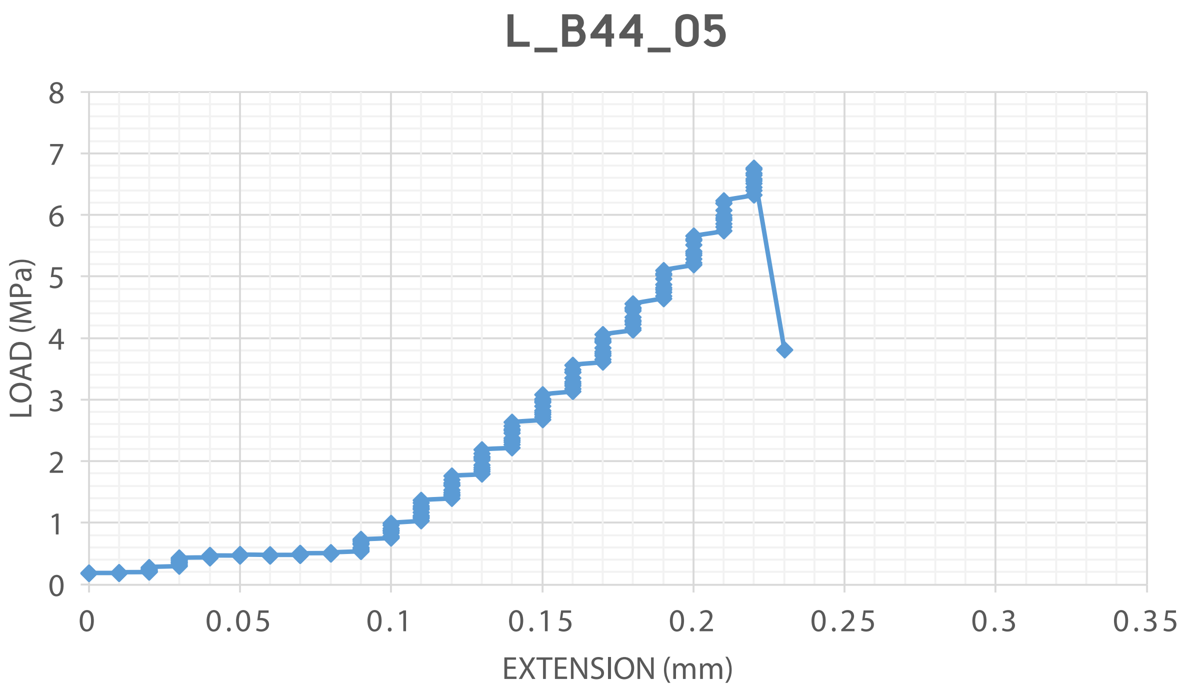
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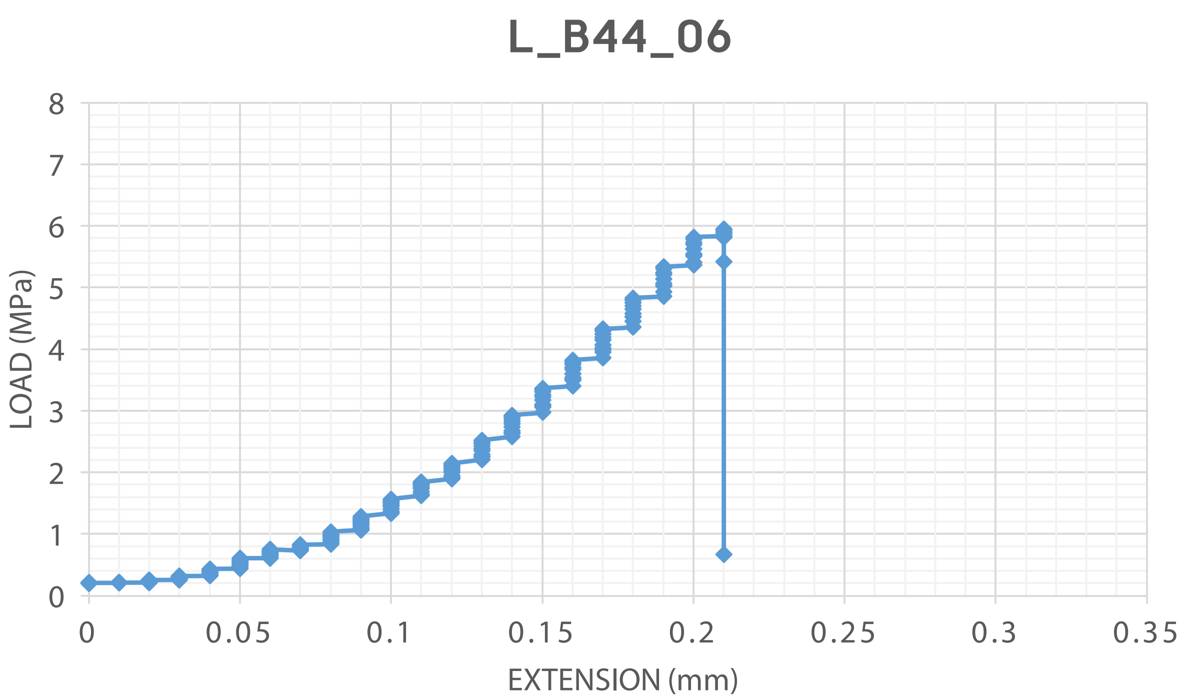
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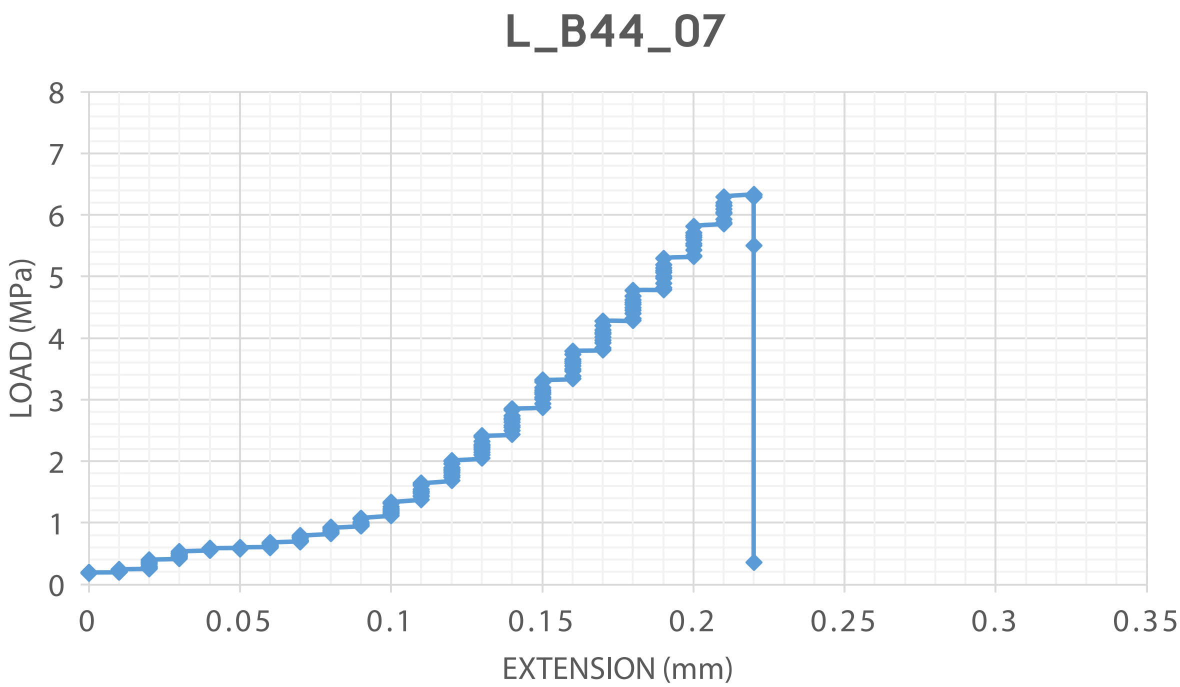
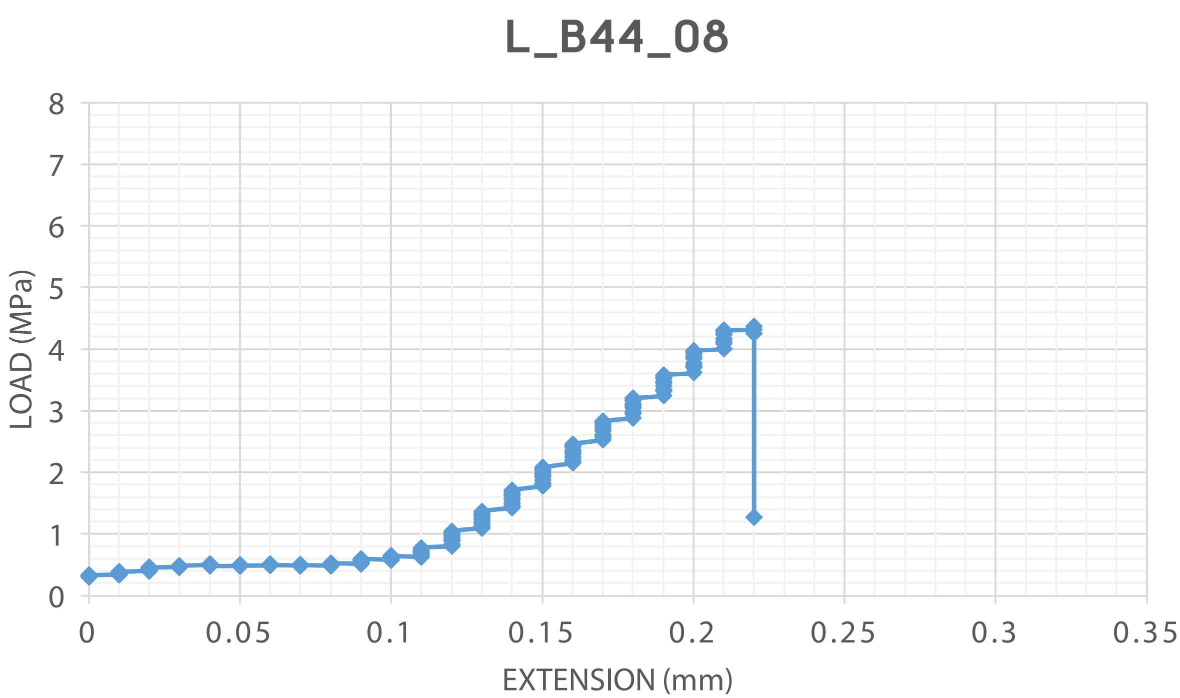
0.35
EXTENSION (mm)


L_B44_07
LOAD (MPa)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B44_08
LOAD (MPa)
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1
2
3
4
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6
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8
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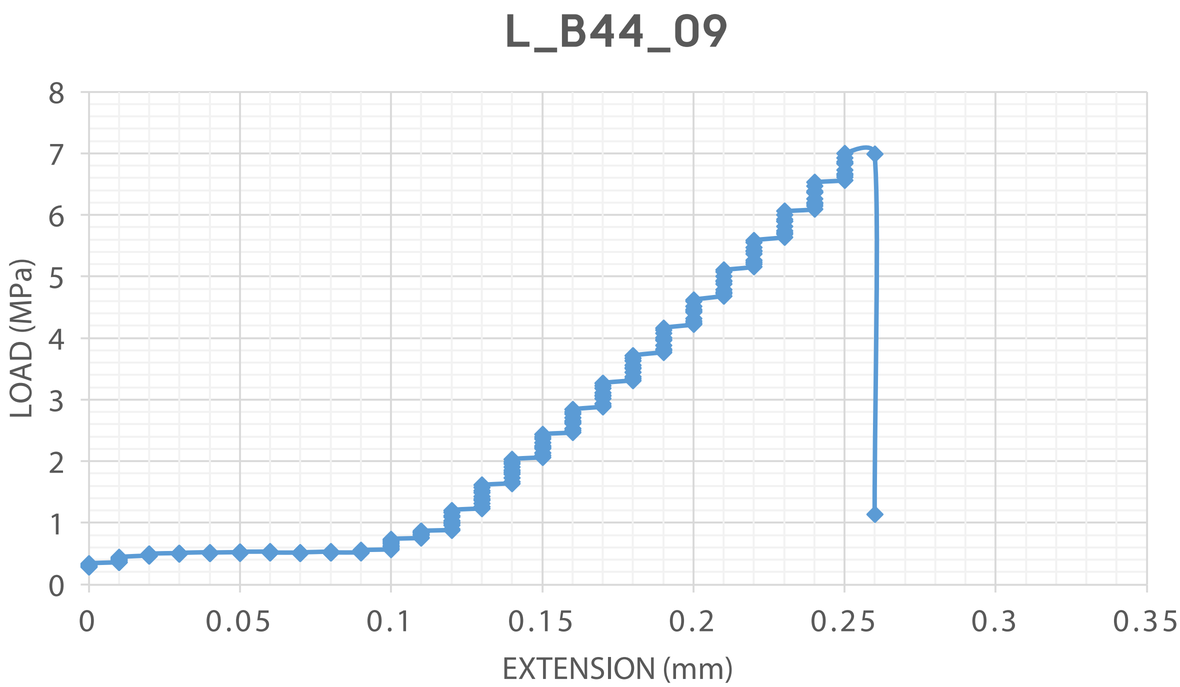
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B44_09
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
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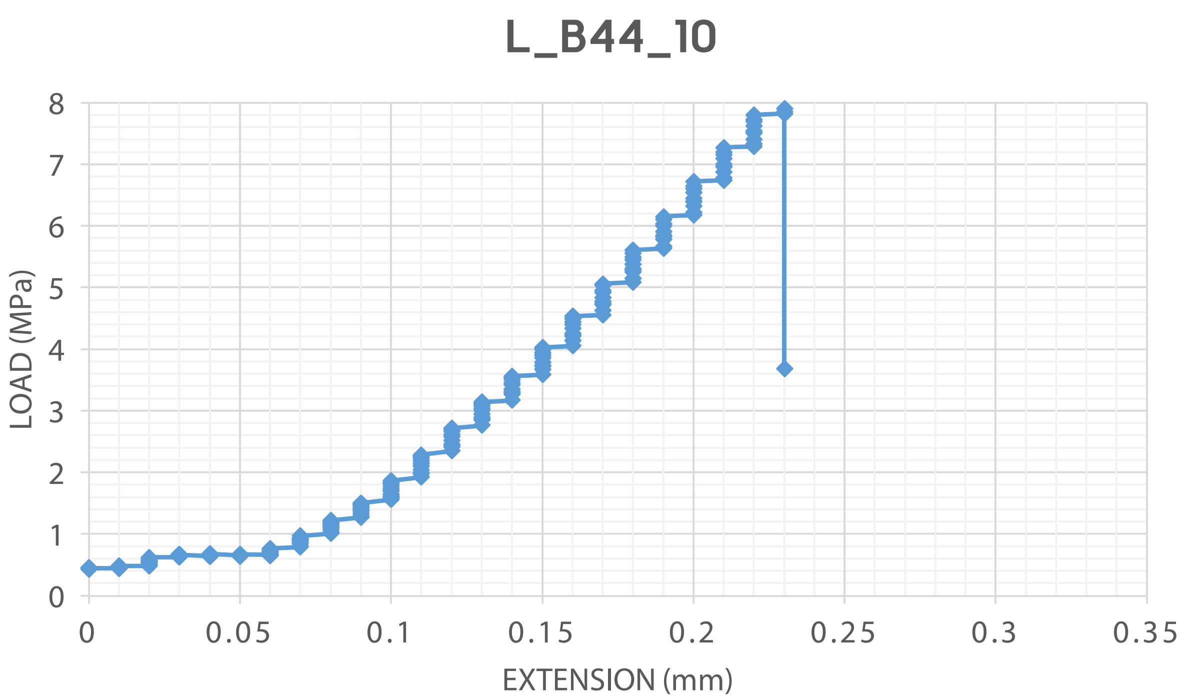
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B44_10
LOAD (MPa)
EXTENSION (mm)
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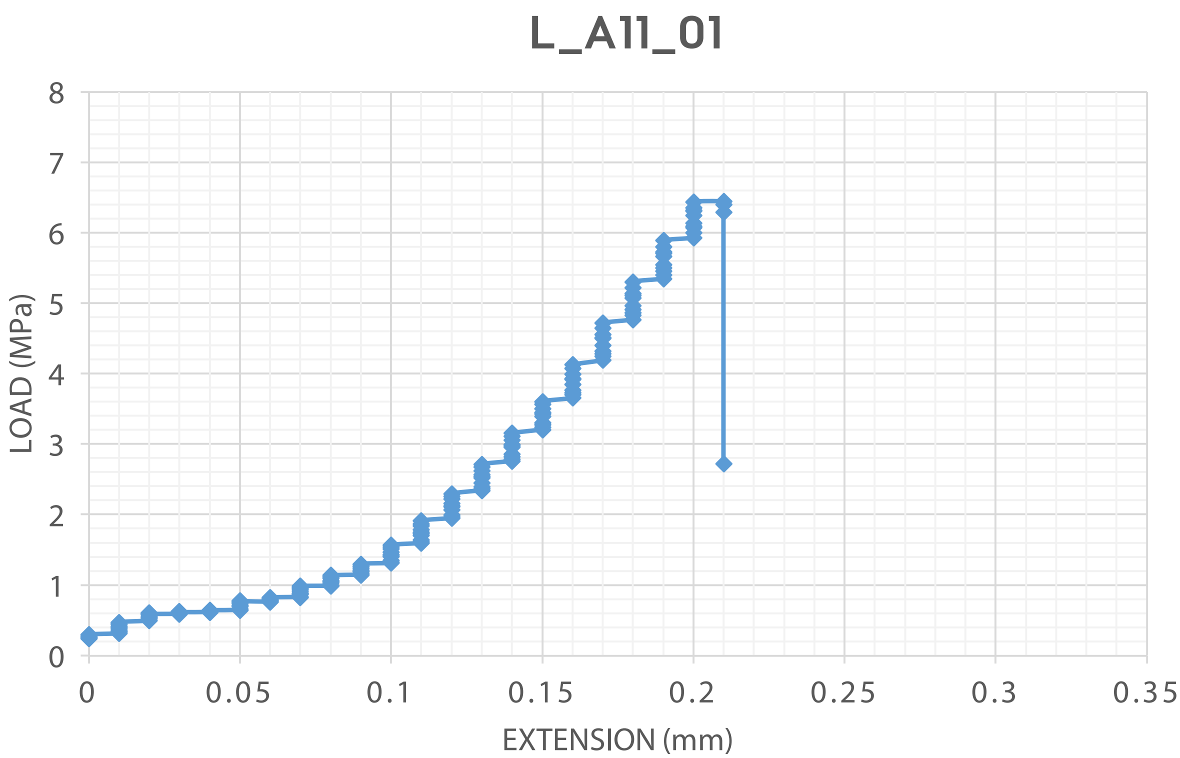
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_A11_01
8
7
6
5
4
3
2
1
0
LOAD (MPa)
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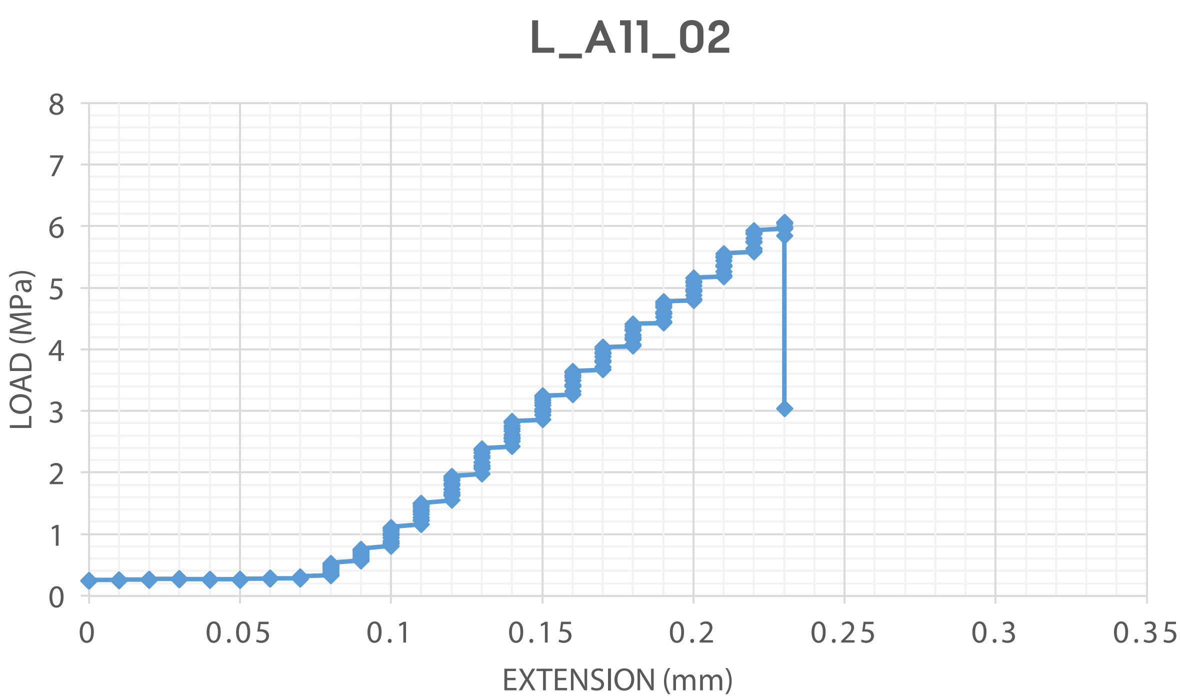
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_A11_02
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7
6
5
4
3
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0
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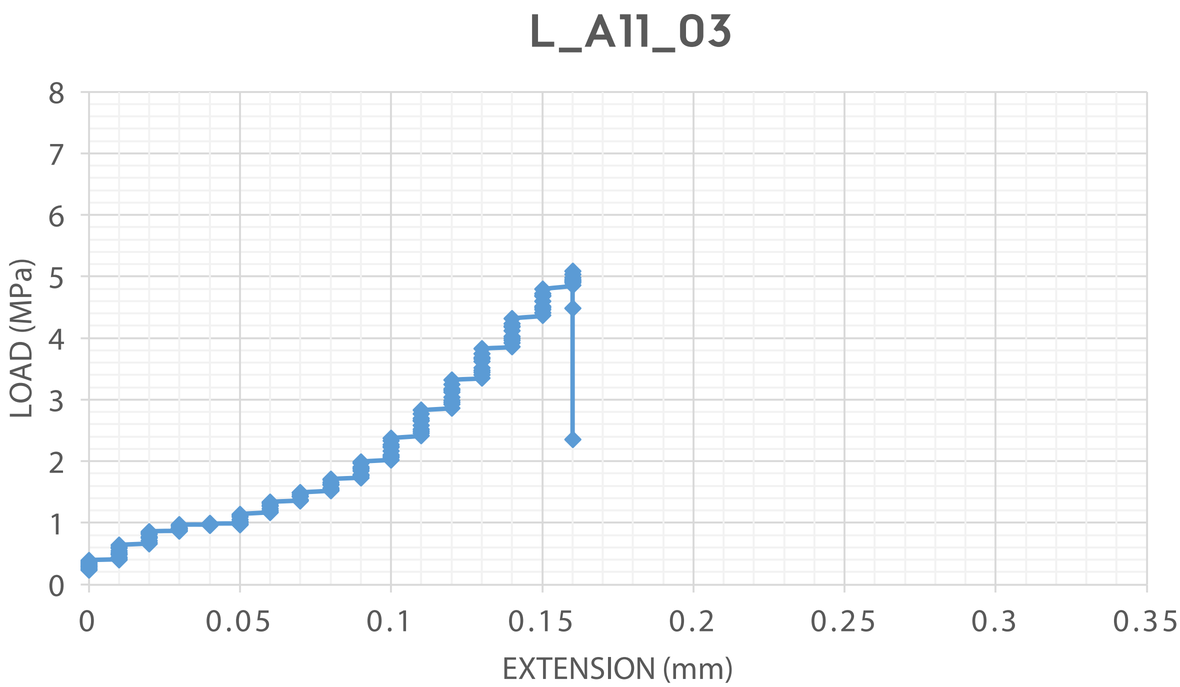
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_A11_03
LOAD (MPa)
EXTENSION (mm)
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7
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0
0.05
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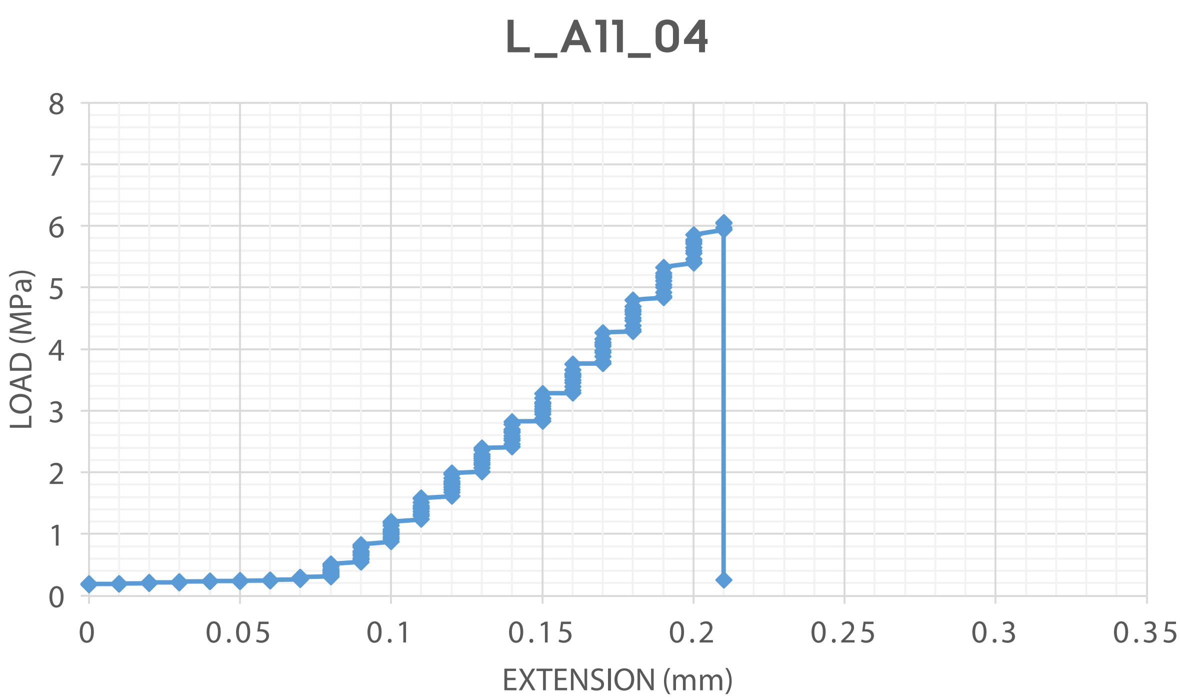
0.15
0.2
0.25
0.3
0.35


L_A11_04
LOAD (MPa)
EXTENSION (mm)
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1
2
3
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5
6
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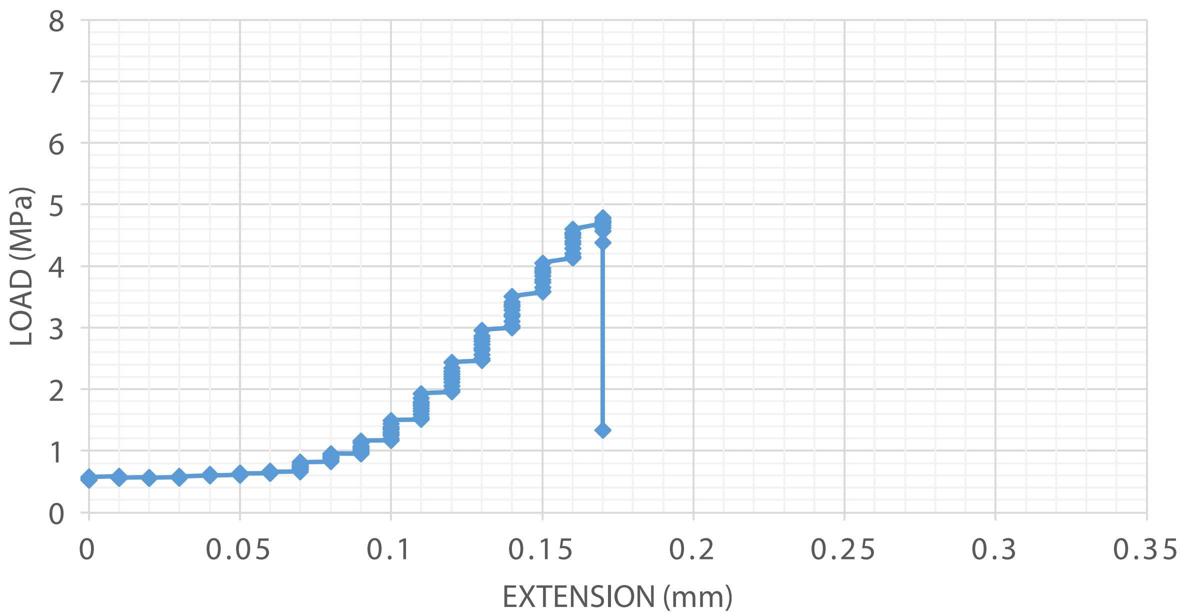
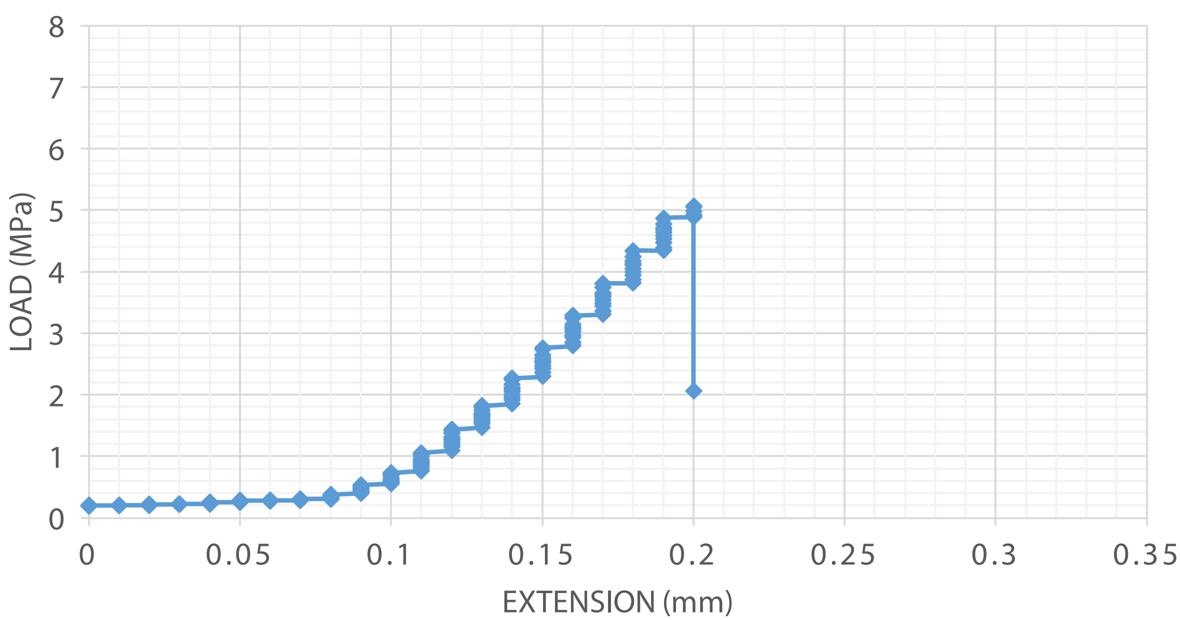
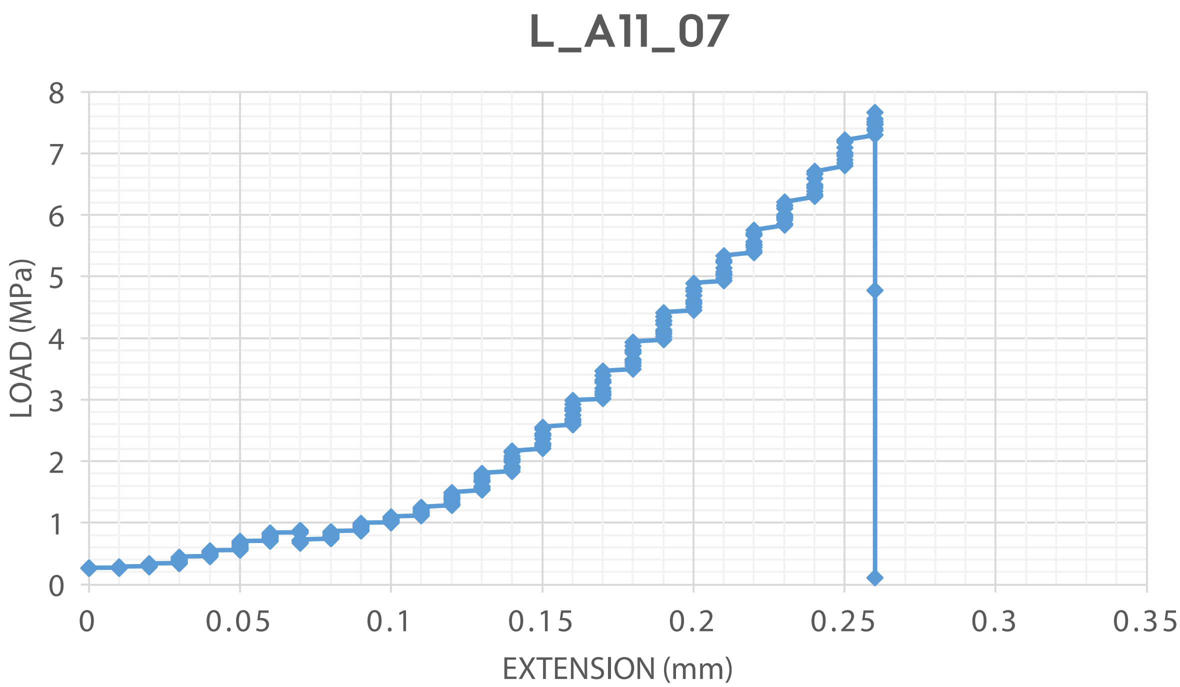
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35

L_A11_05



L_A11_06




L_A11_07
LOAD (MPa)
EXTENSION (mm)
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3
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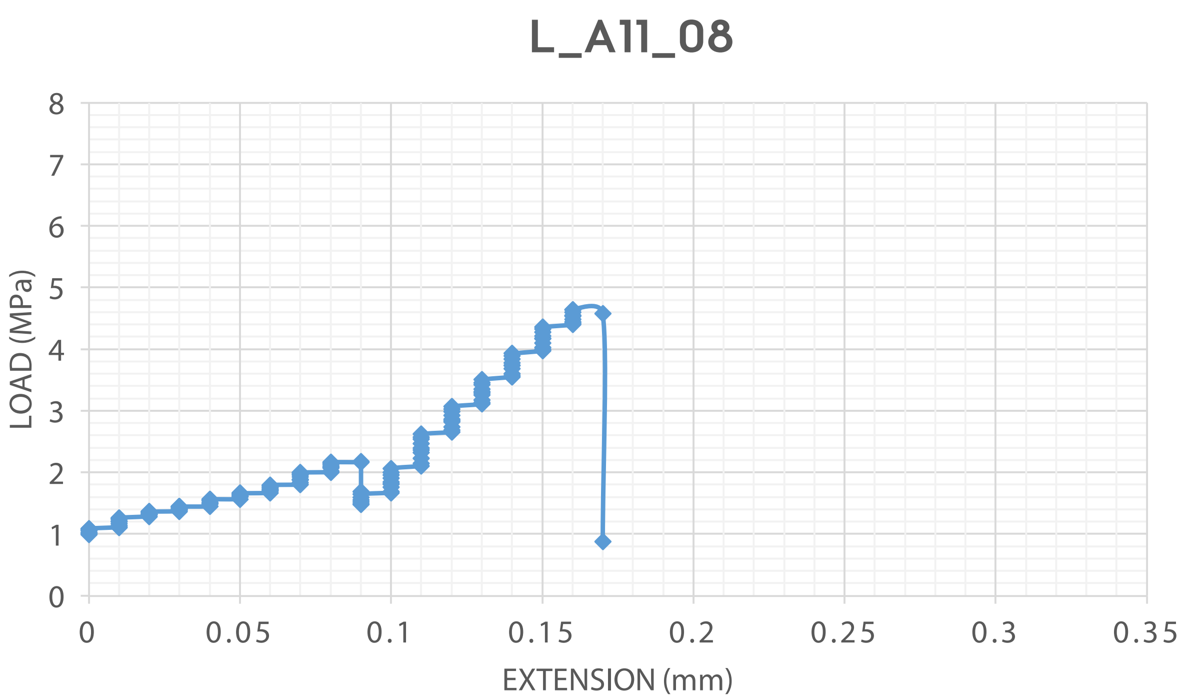
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_A11_08
LOAD (MPa)
EXTENSION (mm)
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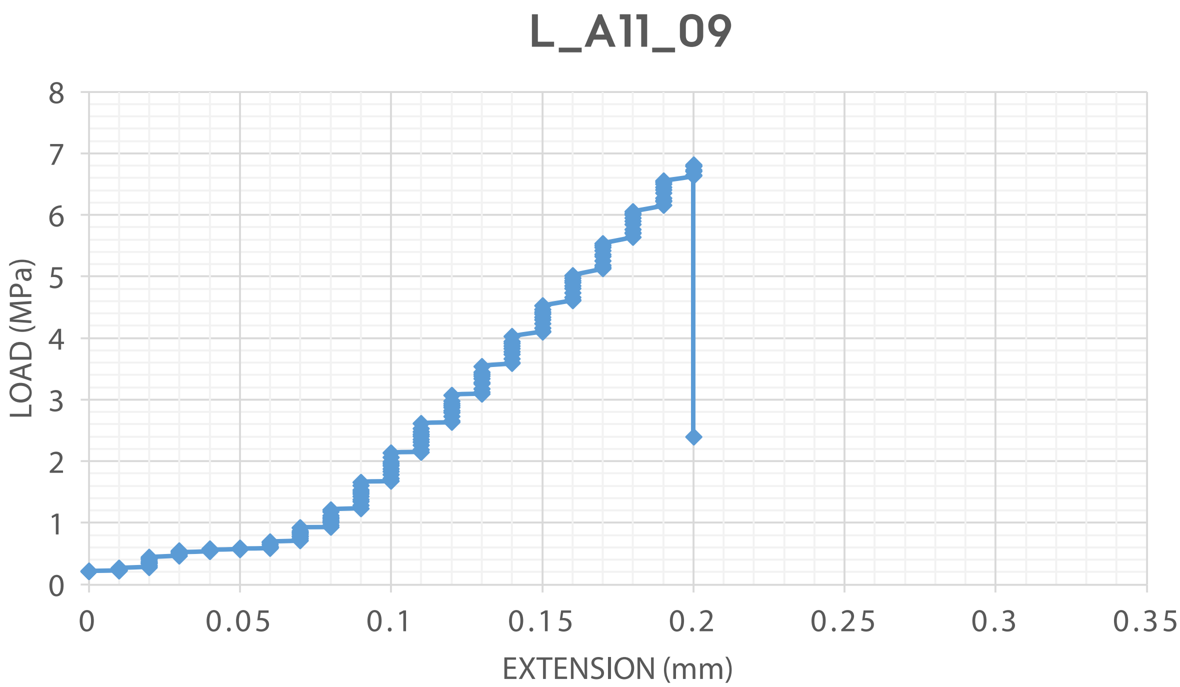
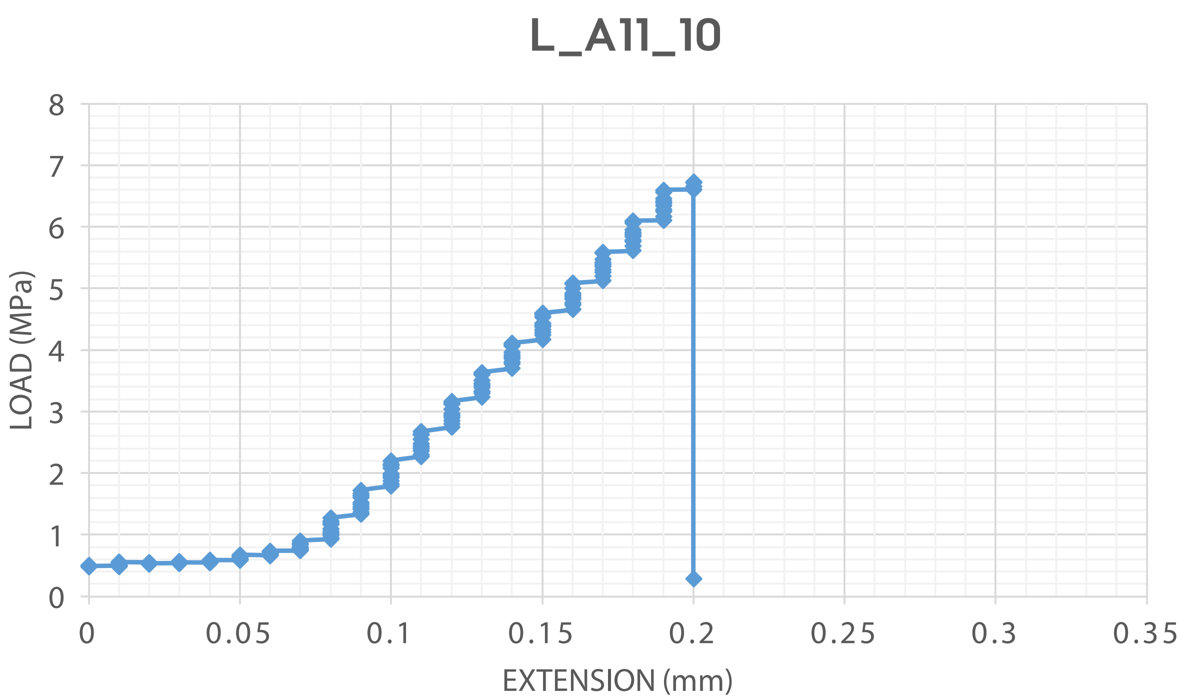
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35



L_A11_09
LOAD (MPa)
8
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)



L_A11_10
LOAD (MPa)
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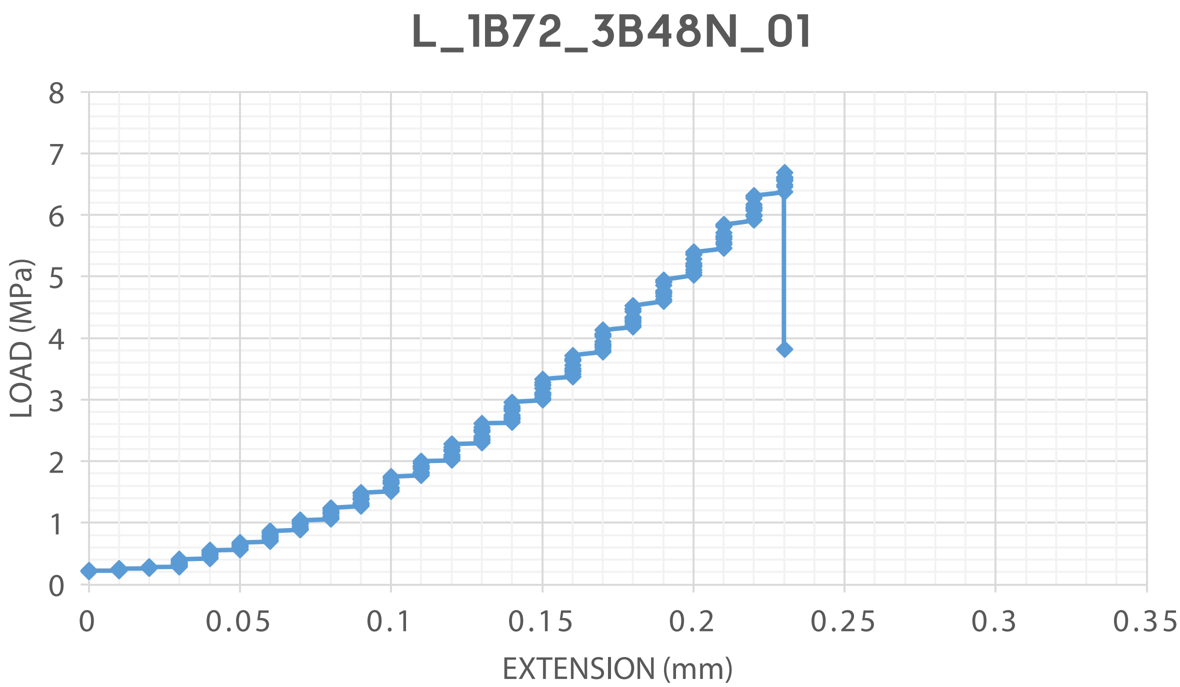
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_1B72_3B48N_01
LOAD (MPa)
EXTENSION (mm)
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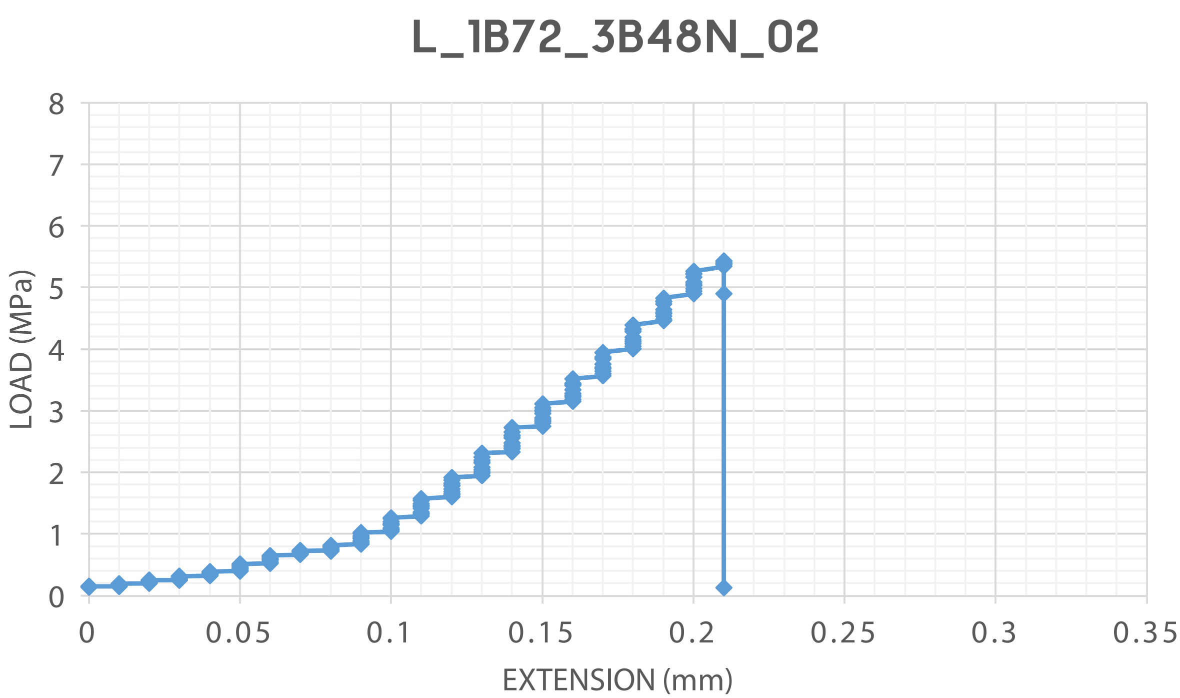
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_02
LOAD (MPa)
EXTENSION (mm)
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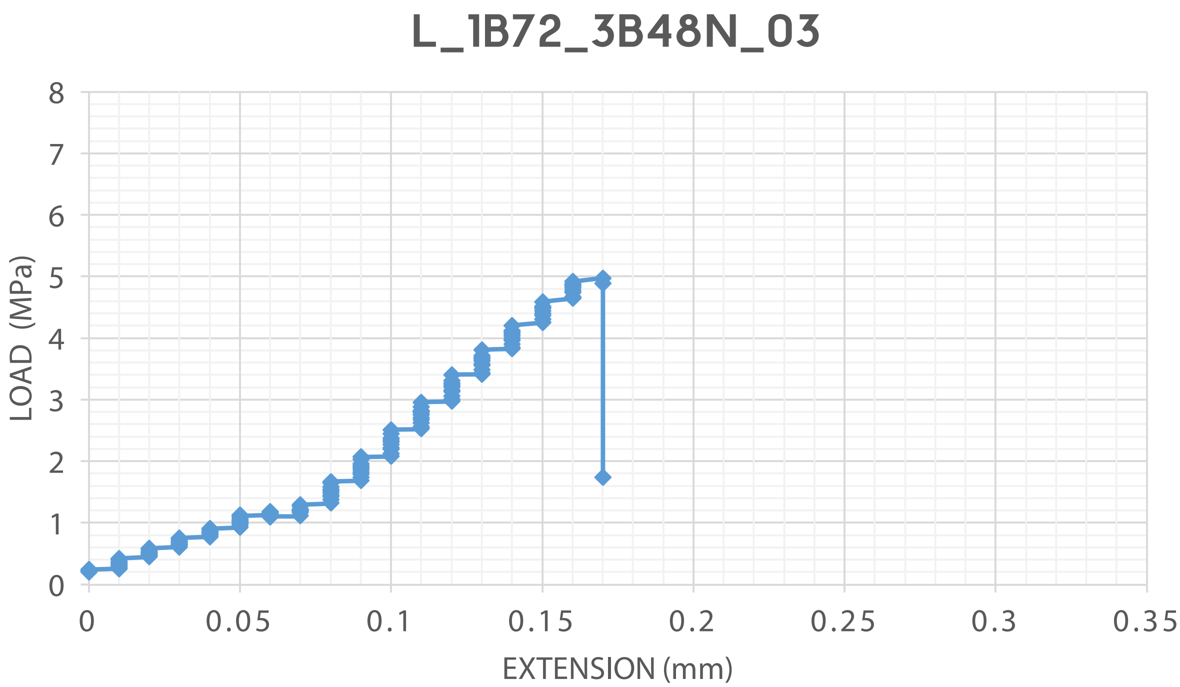
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_03
LOAD (MPa)
EXTENSION (mm)
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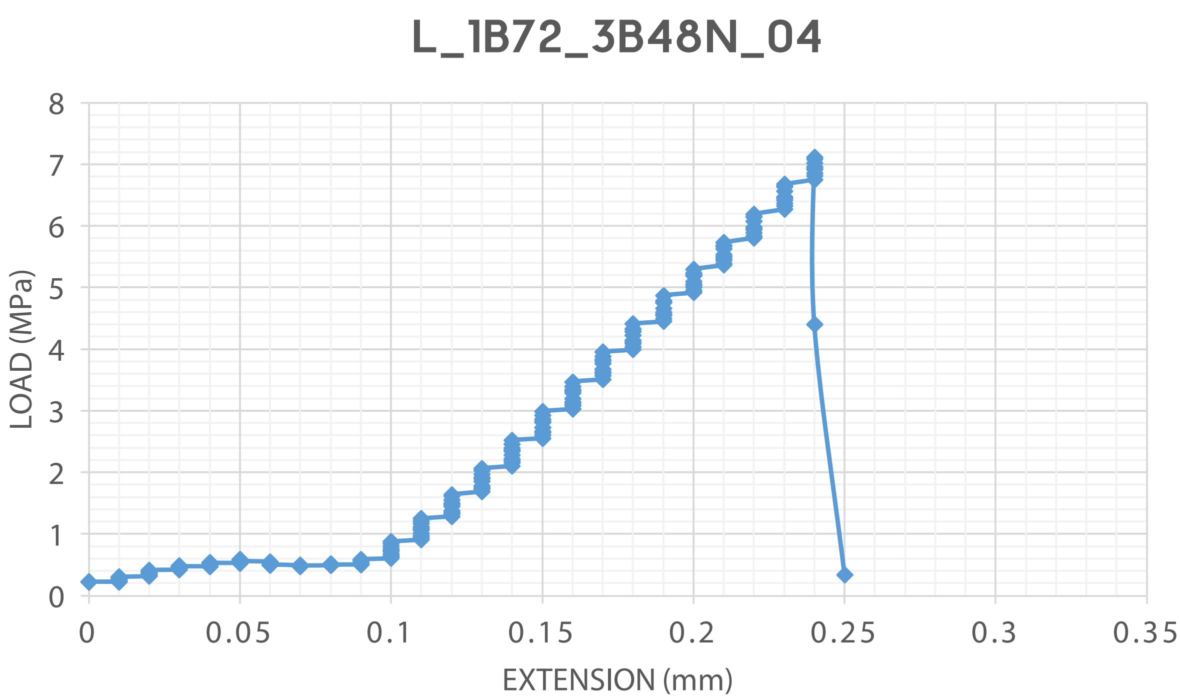
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_04
LOAD (MPa)
EXTENSION (mm)
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1
2
3
4
5
6
7
8
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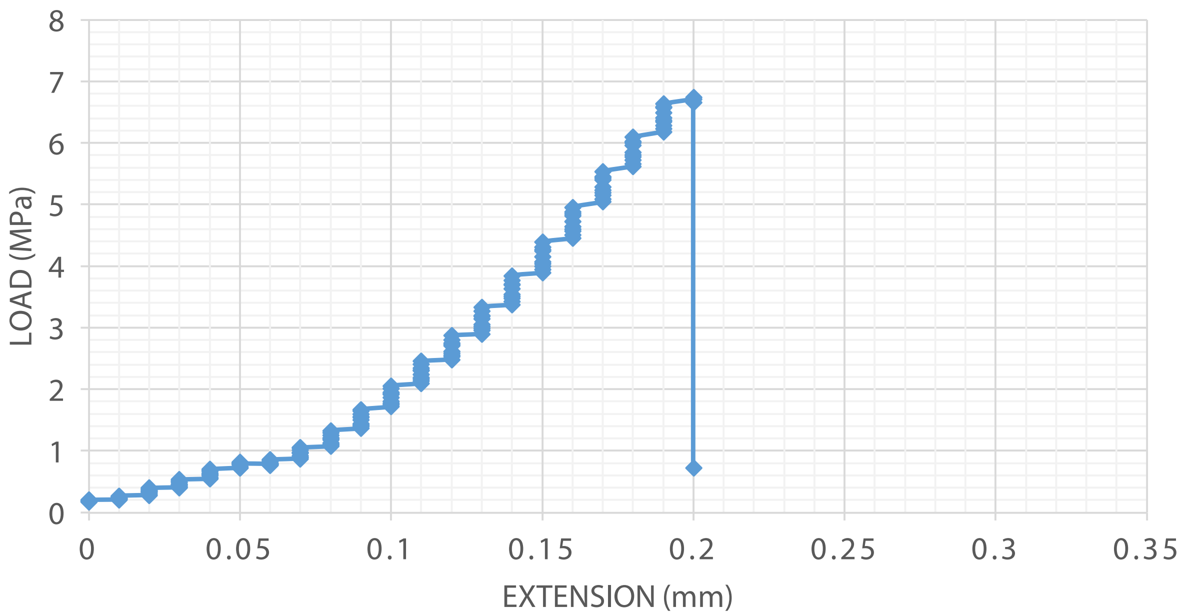
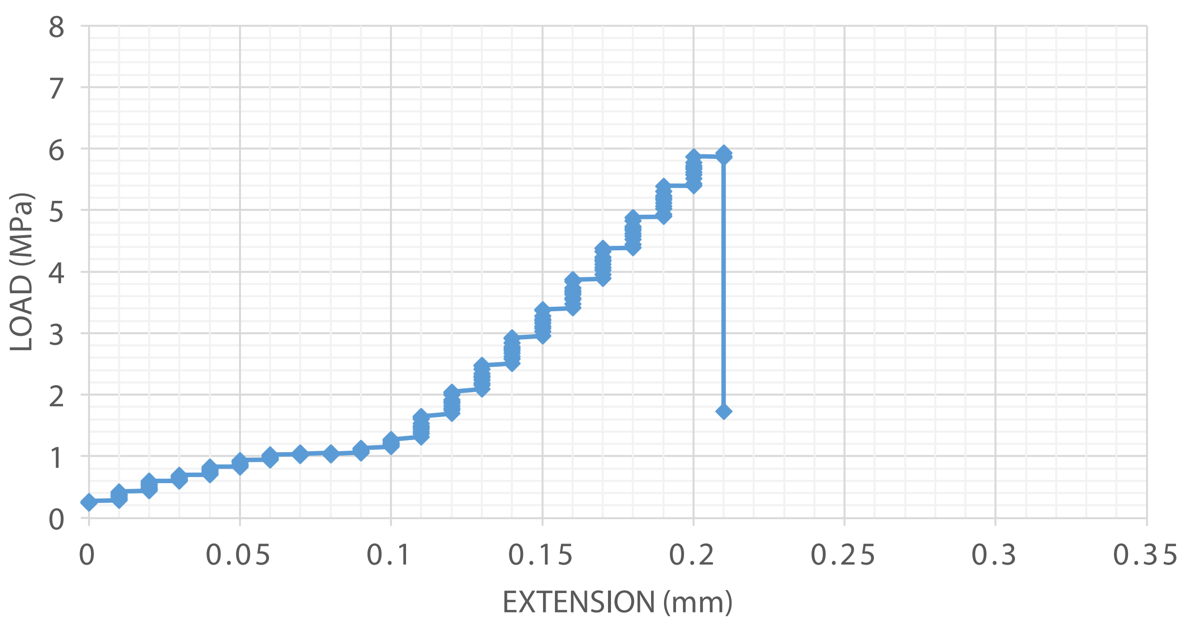
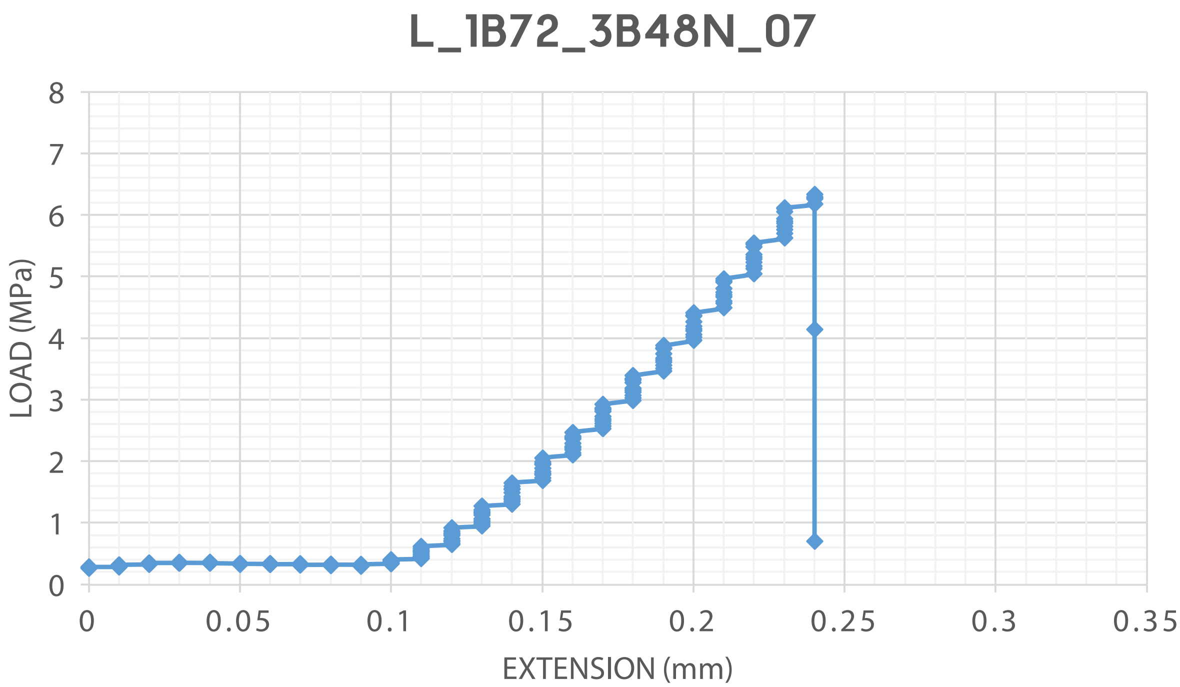
0.05
0.1
0.15
0.2
0.25
0.3
0.35

L_1B72_3B48N_05



L_1B72_3B48N_06




L_1B72_3B48N_07
LOAD (MPa)
EXTENSION (mm)
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3
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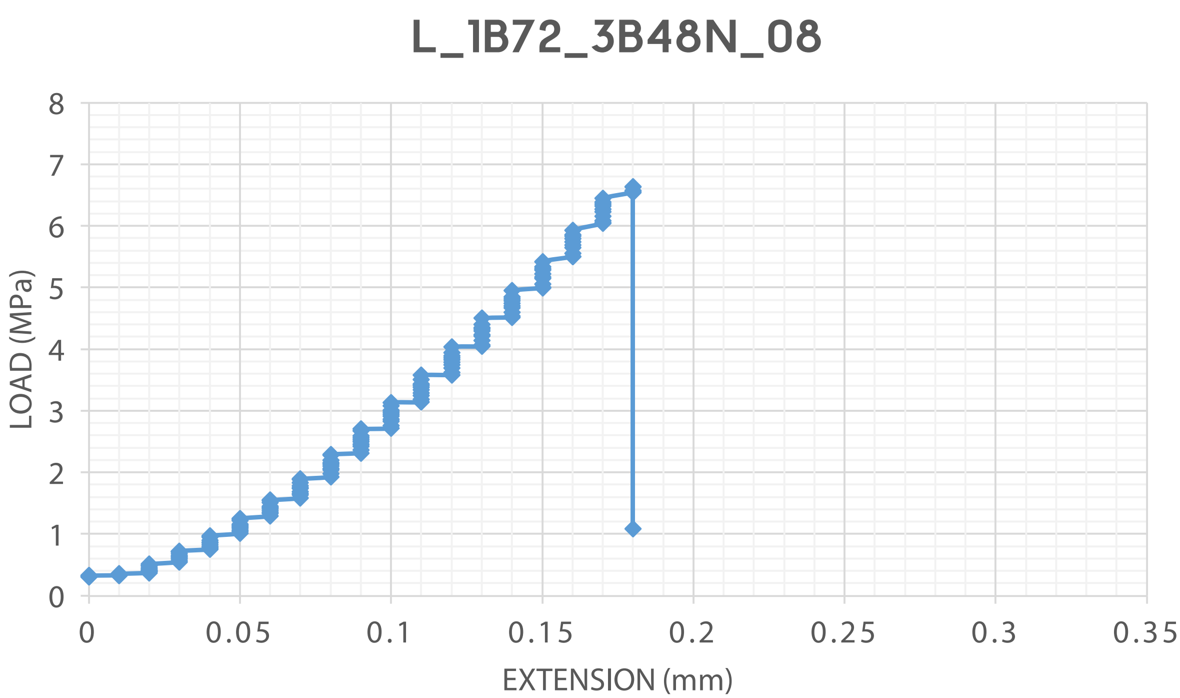
7
8
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_08
LOAD (MPa)
EXTENSION (mm)
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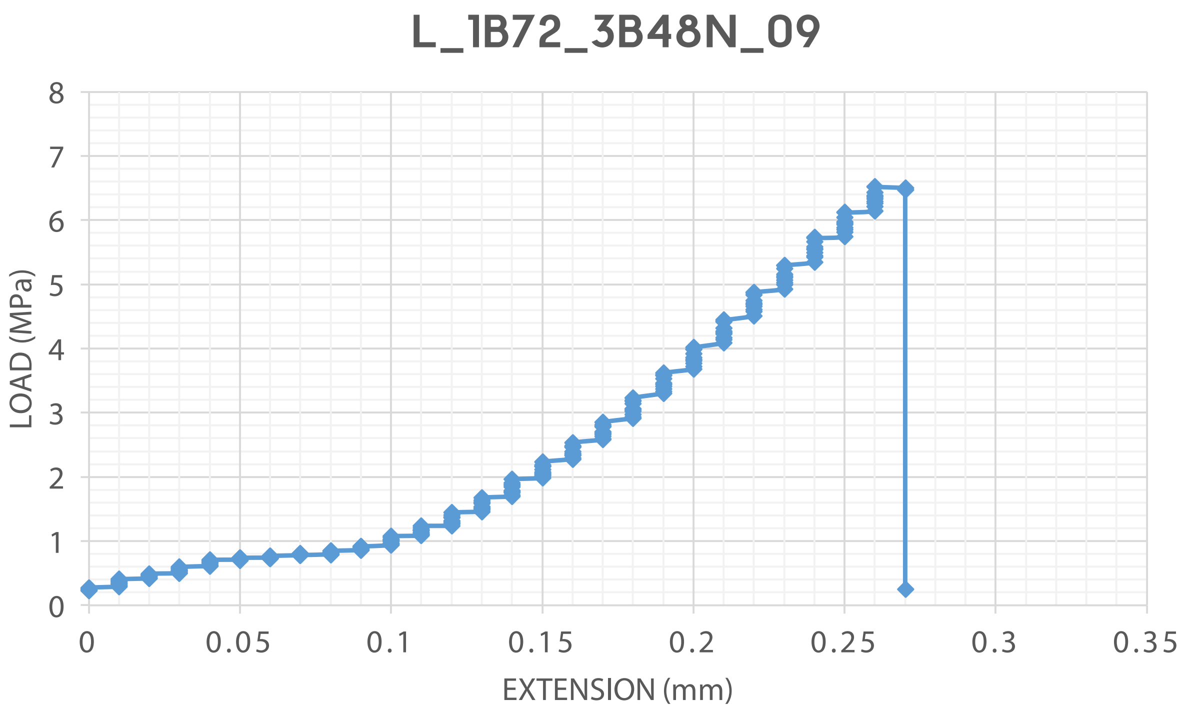
3
4
5
6
7
8
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_09
LOAD (MPa)
EXTENSION (mm)
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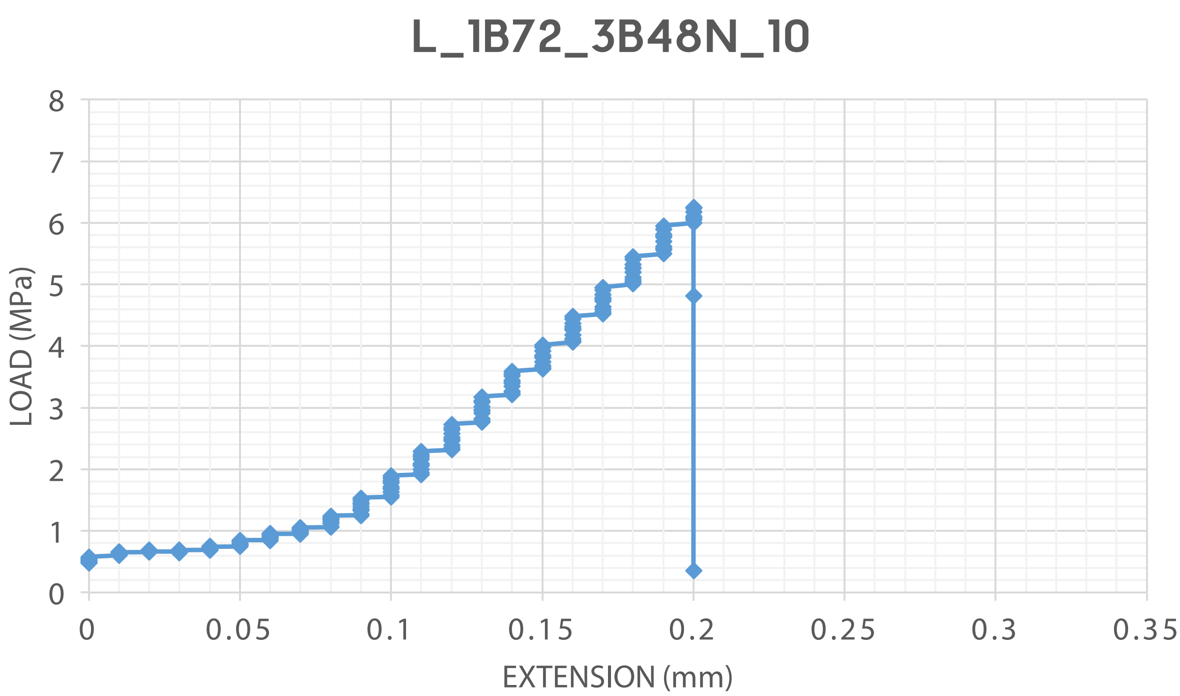
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_10
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EXTENSION (mm)
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3
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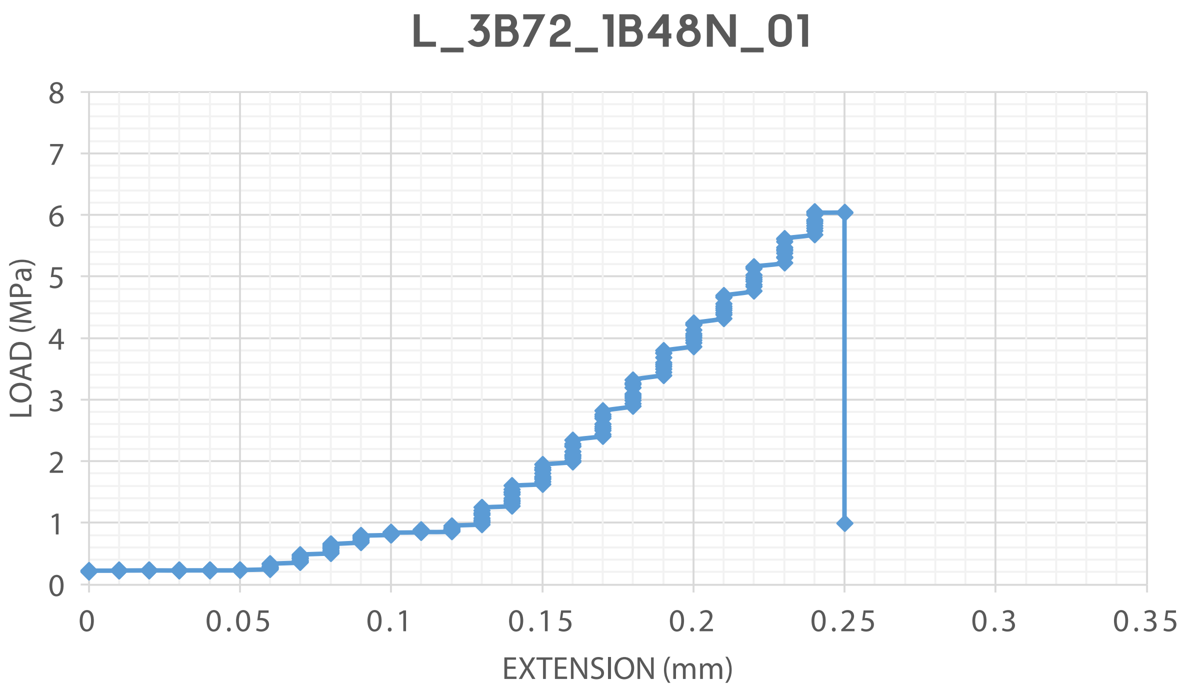
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_3B72_1B48N_01
LOAD (MPa)
EXTENSION (mm)
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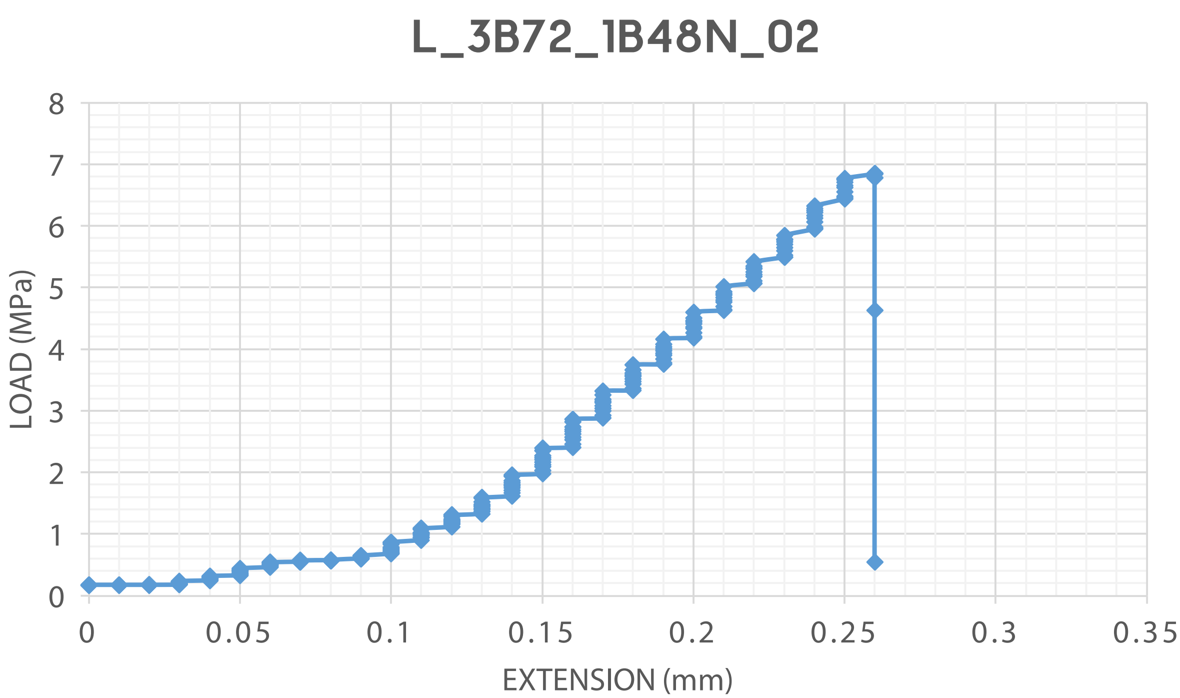
5
6
7
8
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_3B72_1B48N_02
LOAD (MPa)
EXTENSION (mm)
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6
7
8
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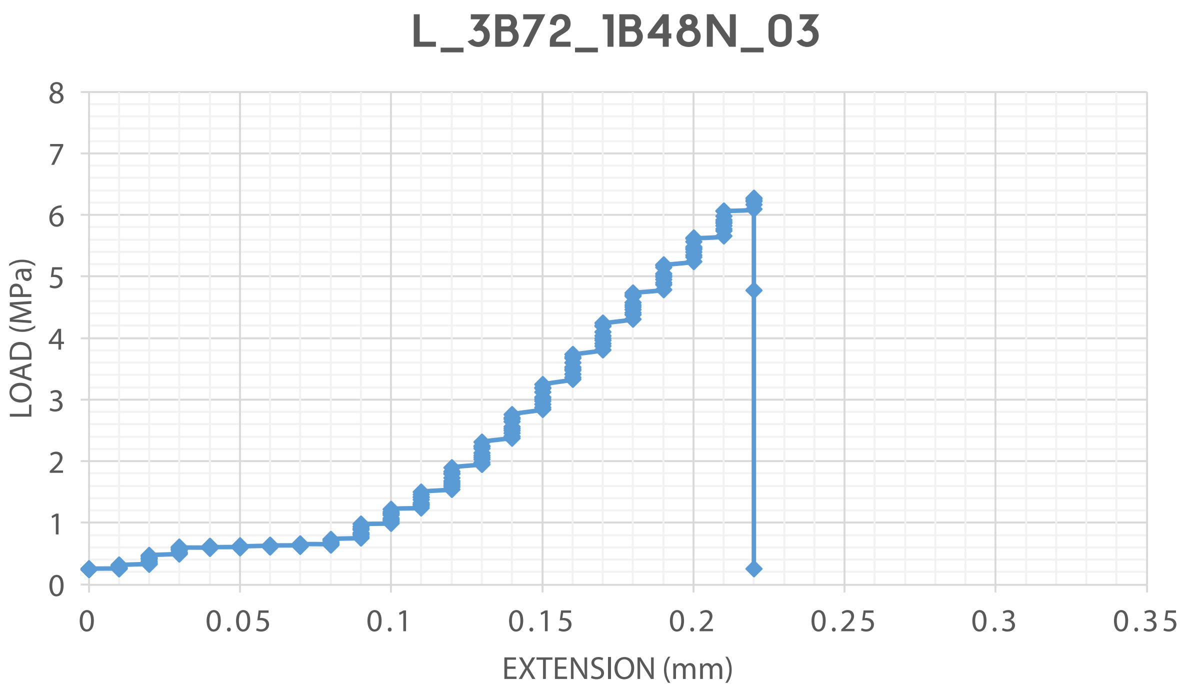
0.15
0.2
0.25
0.3
0.35


L_3B72_1B48N_03
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2
3
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6
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8
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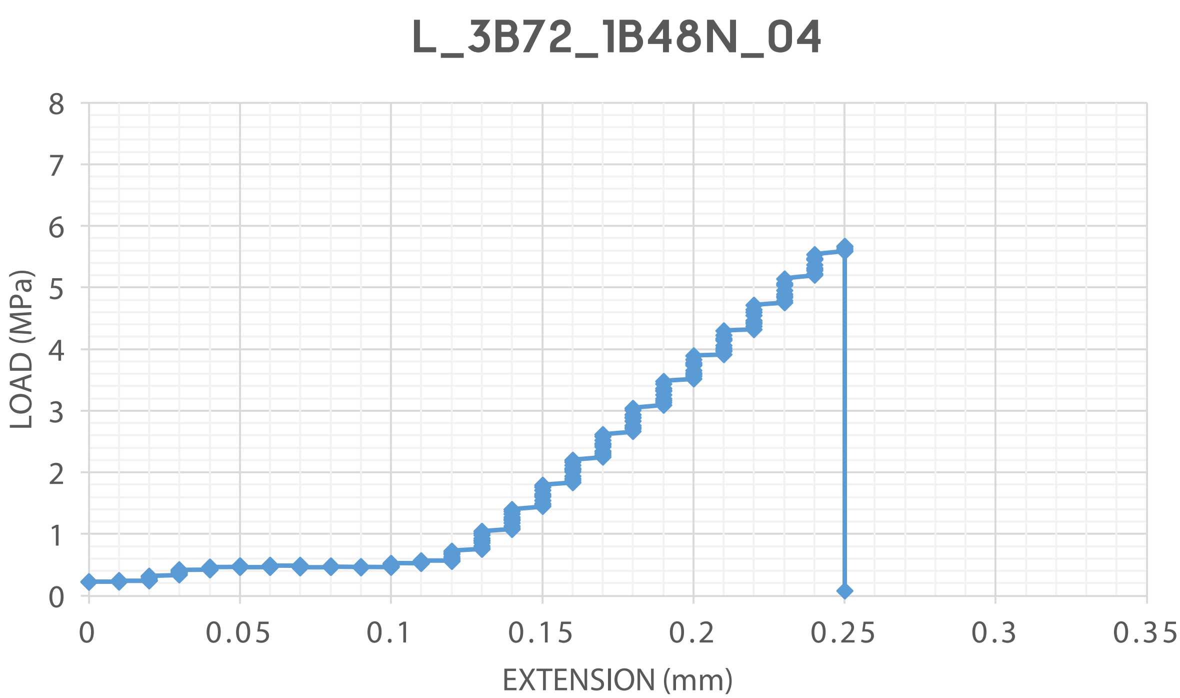
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_3B72_1B48N_04
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2
3
4
5
6
7
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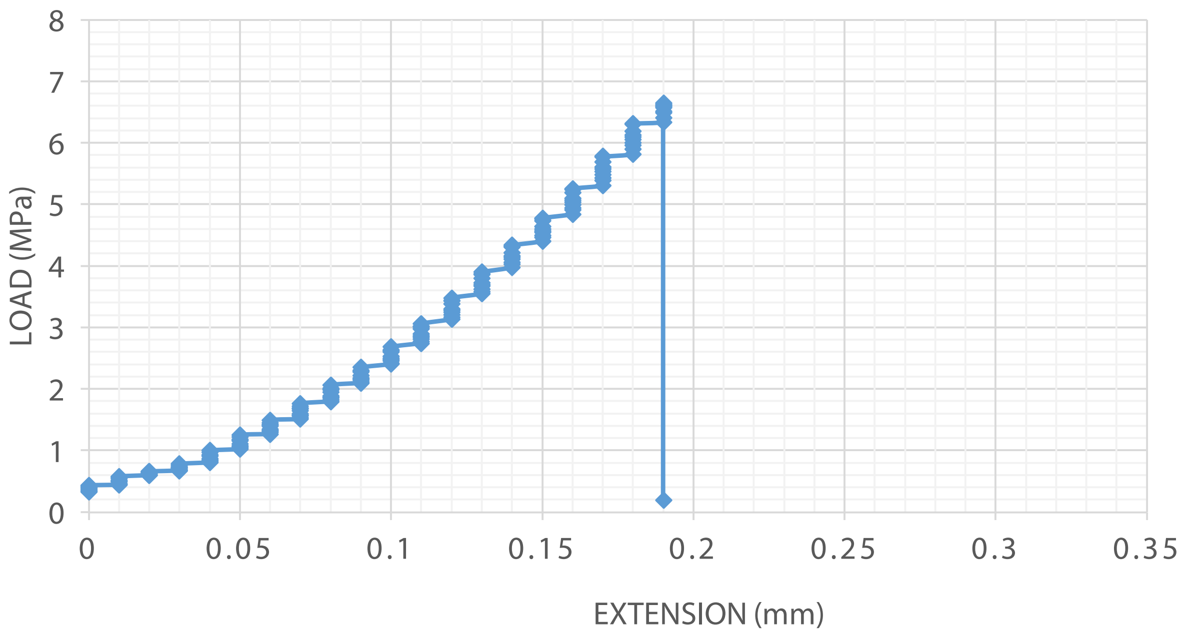
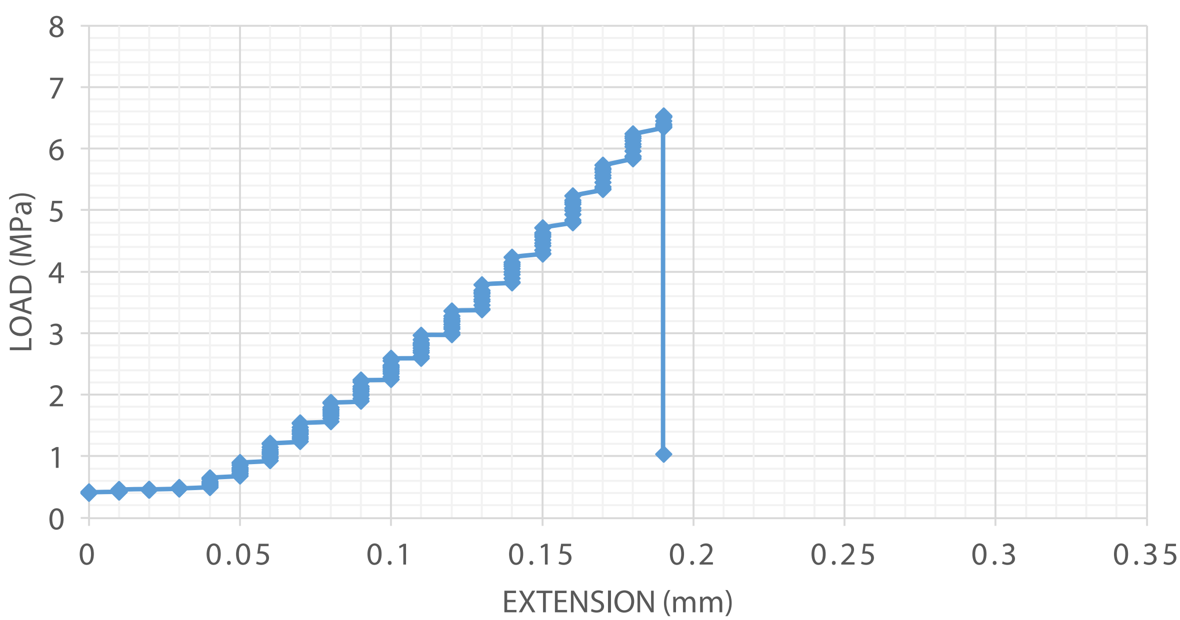
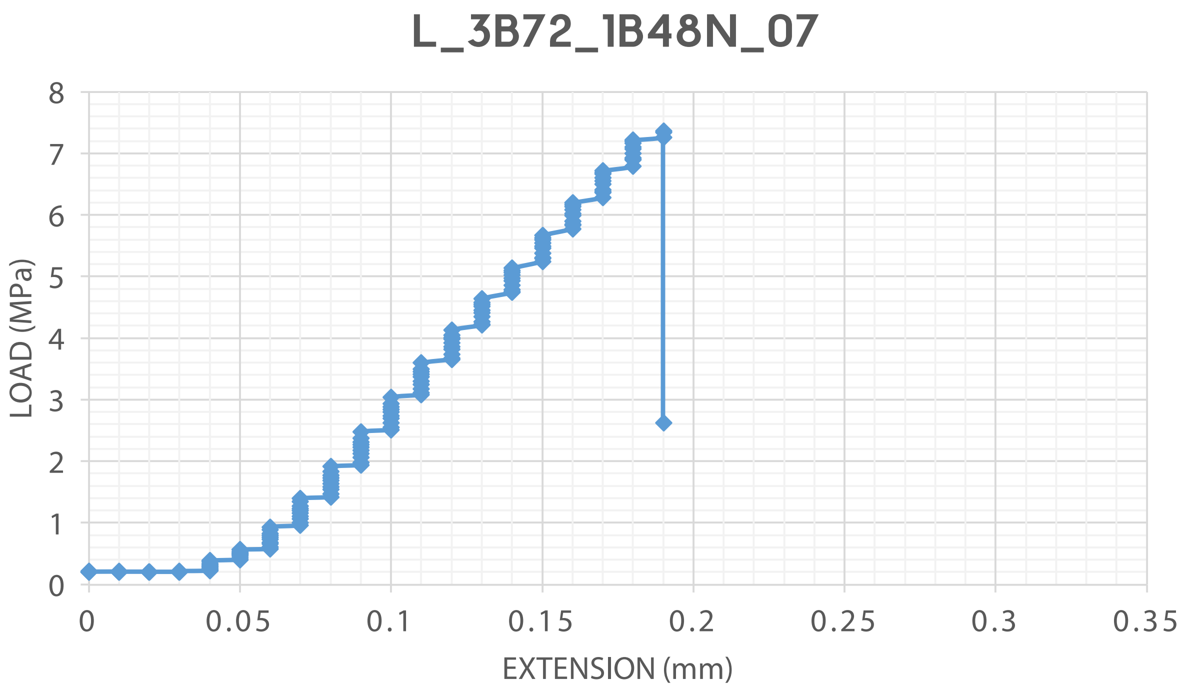
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)

L_3B72_1B48N_05



L_3B72_1B48N_06




L_3B72_1B48N_07
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7
6
5
4
3
2
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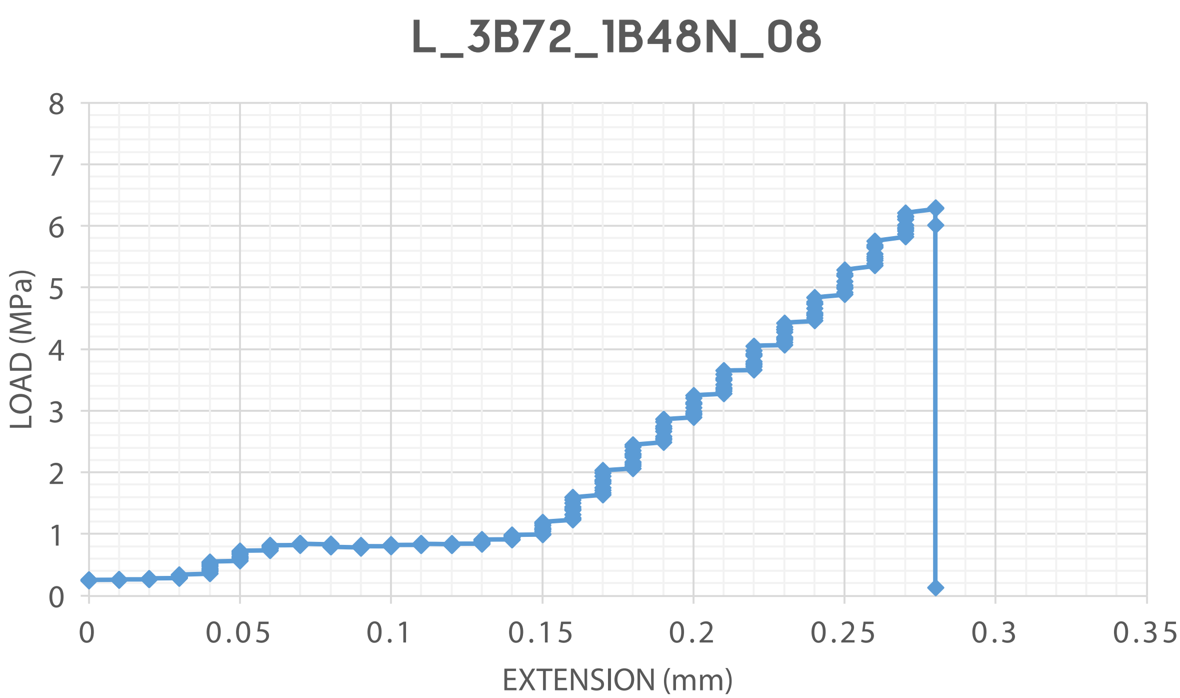
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_3B72_1B48N_08
LOAD (MPa)
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7
6
5
4
3
2
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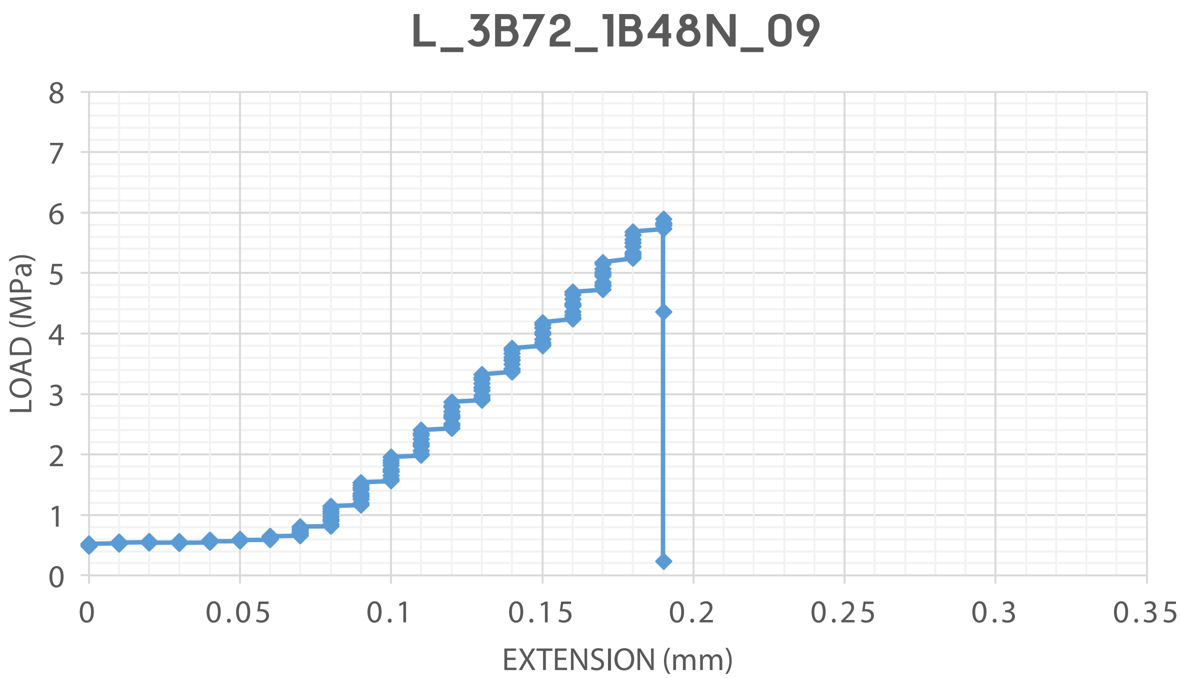
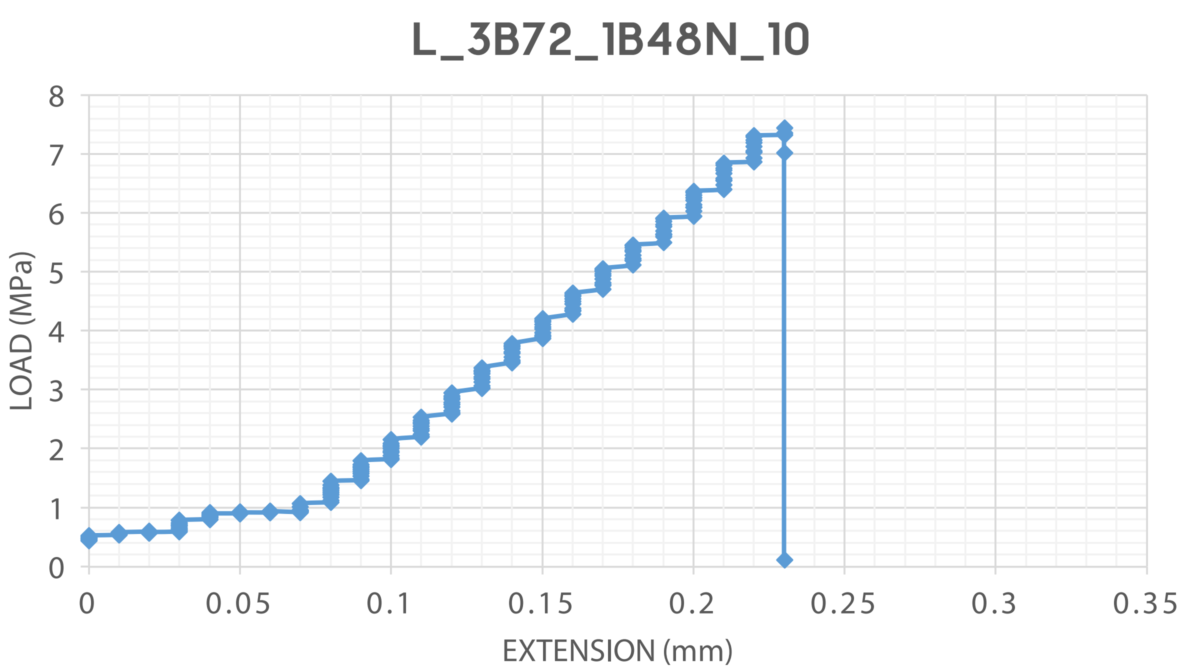
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_3B72_1B48N_09
LOAD (MPa)
8
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_3B72_1B48N_10
LOAD (MPa)
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7
6
5
4
3
2
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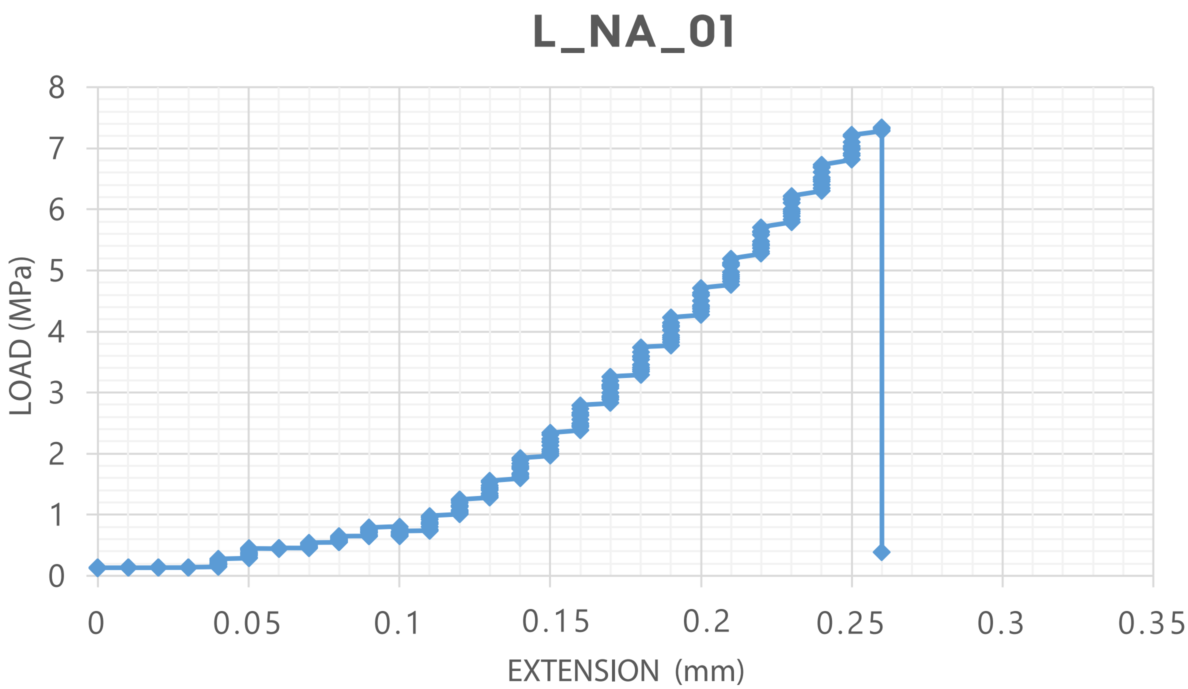
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_NA_01
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2
3
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8
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0.1
0.15
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0.25
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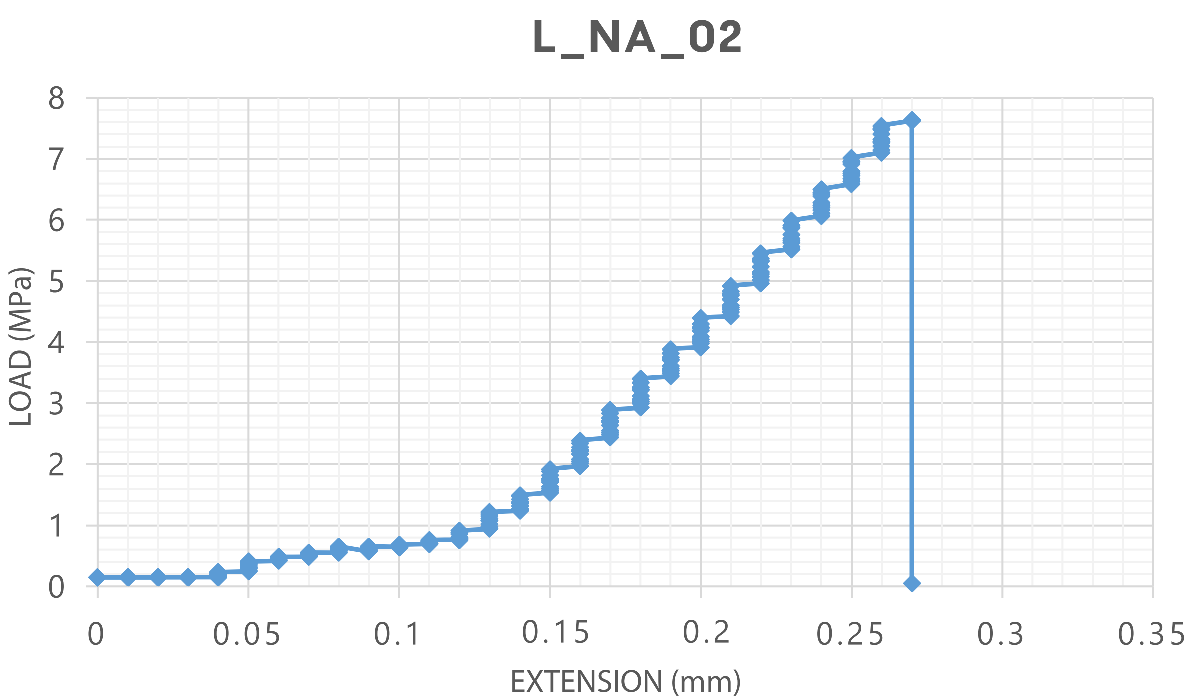
0.35
EXTENSION (mm)


L_NA_02
LOAD (MPa)
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3
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6
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0.1
0.15
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0.25
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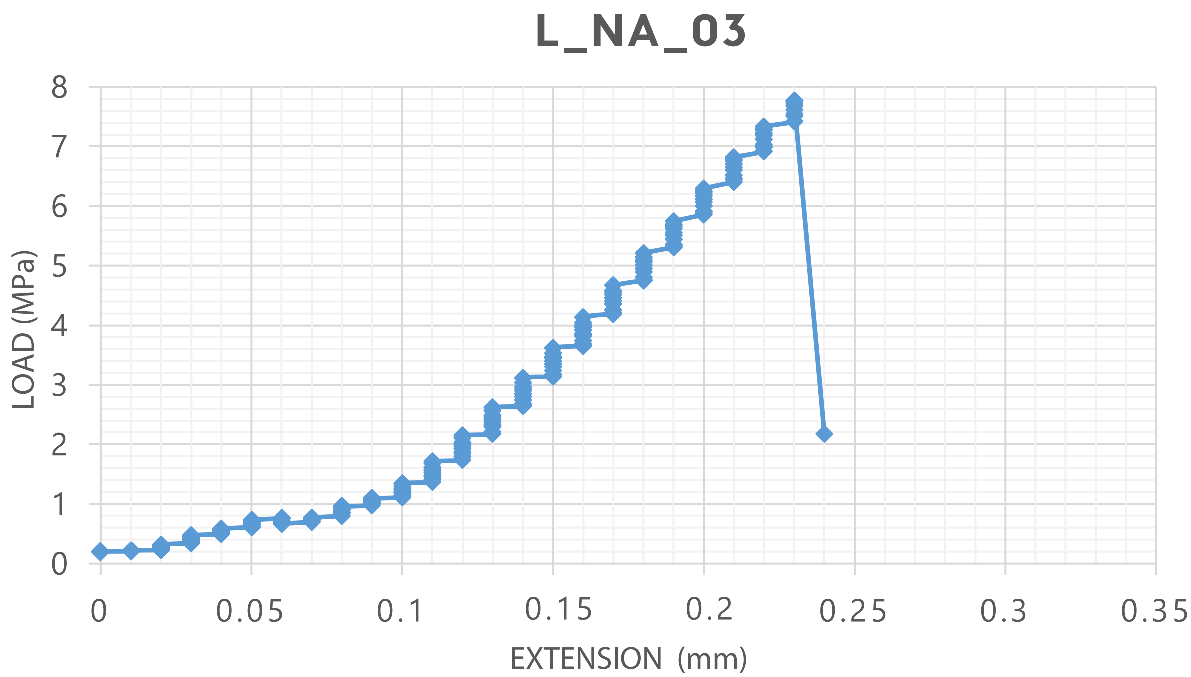
0.35
EXTENSION (mm)


L_NA_03
LOAD (MPa)
EXTENSION (mm)
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0.15
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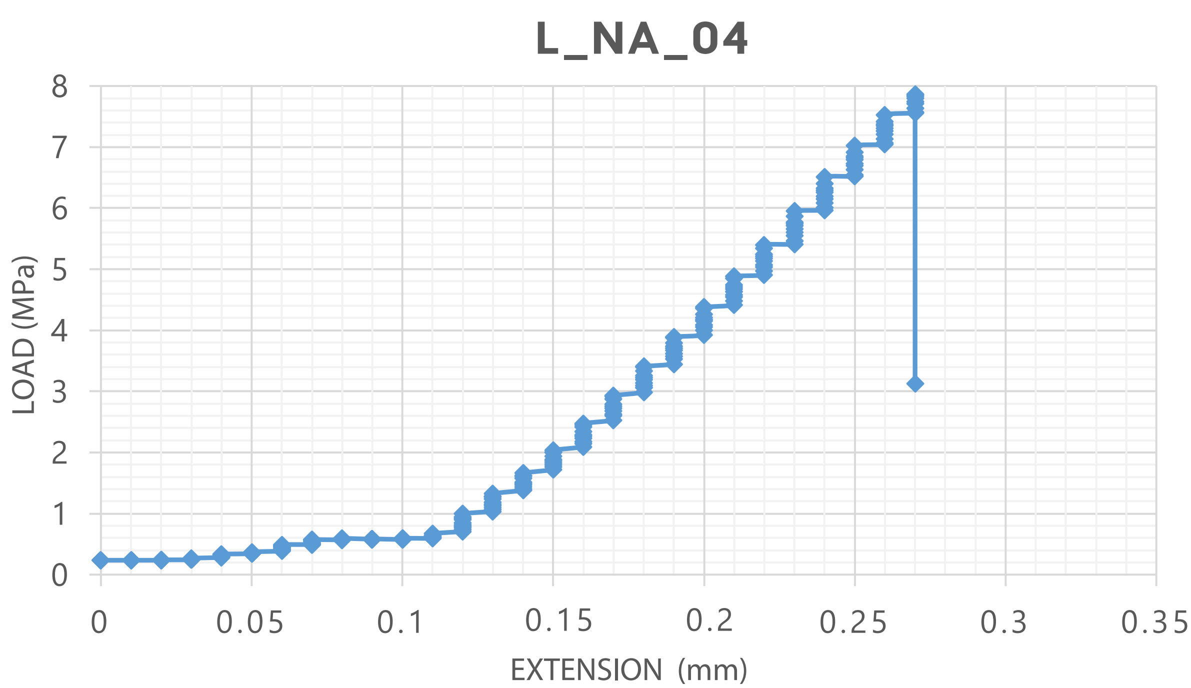
0.25
0.3
0.35


L_NA_04
LOAD (MPa)
EXTENSION (mm)
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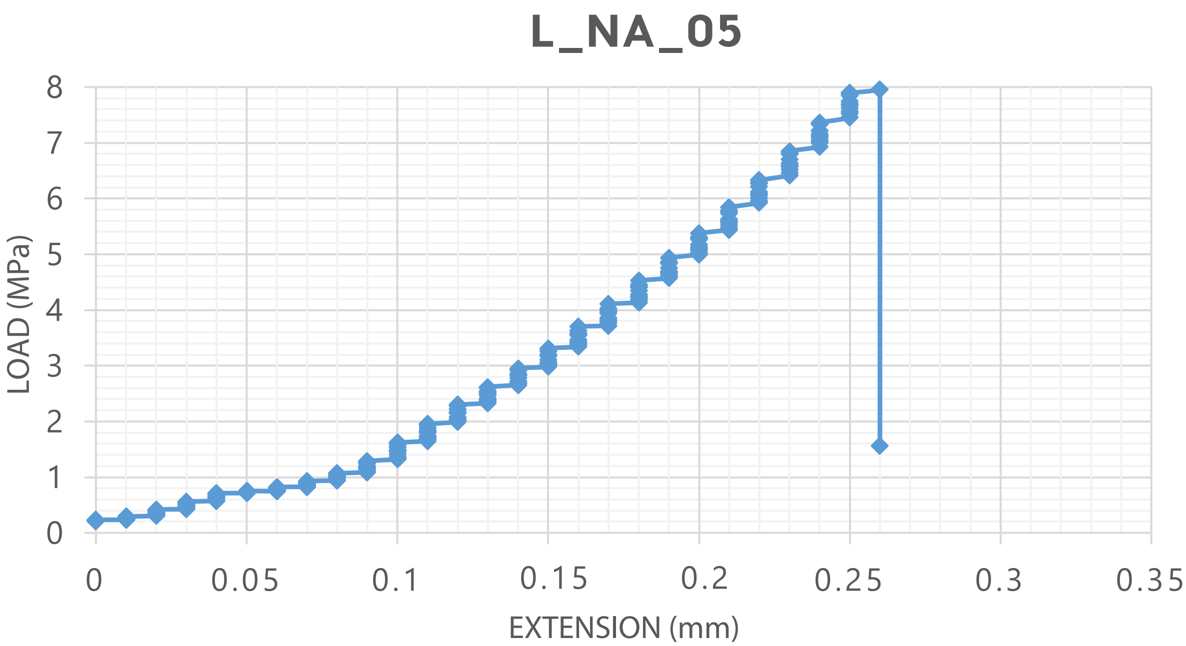
0.15
0.2
0.25
0.3
0.35


L_NA_05
LOAD (MPa)
8
7
6
5
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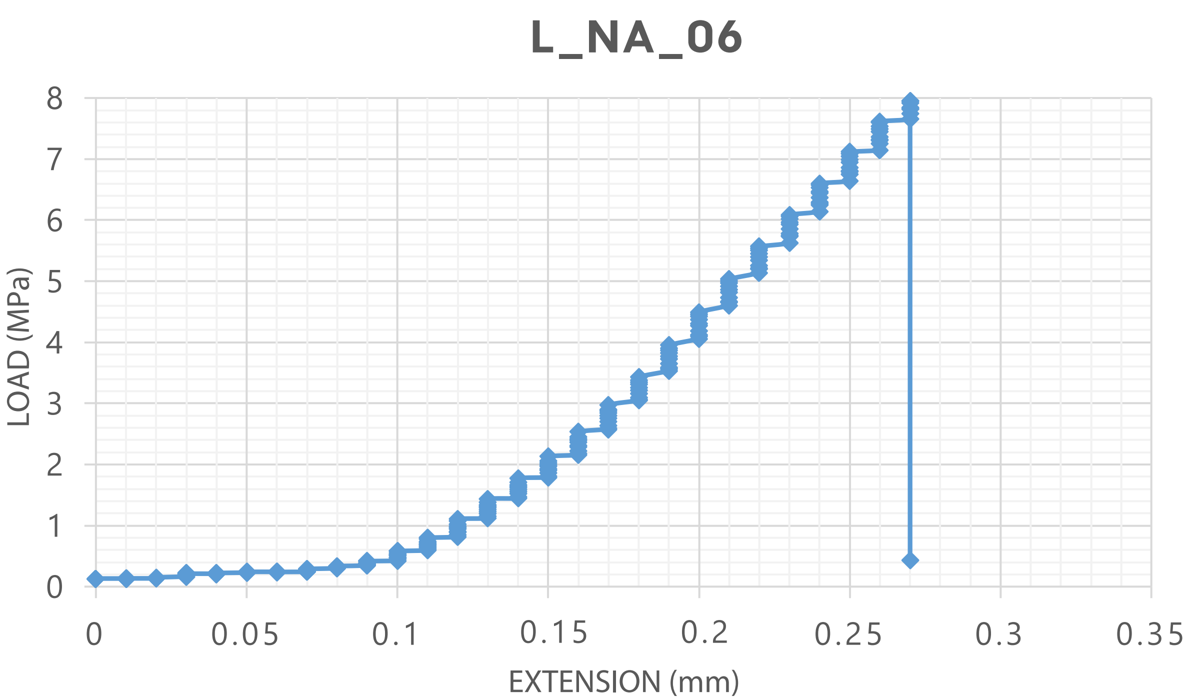
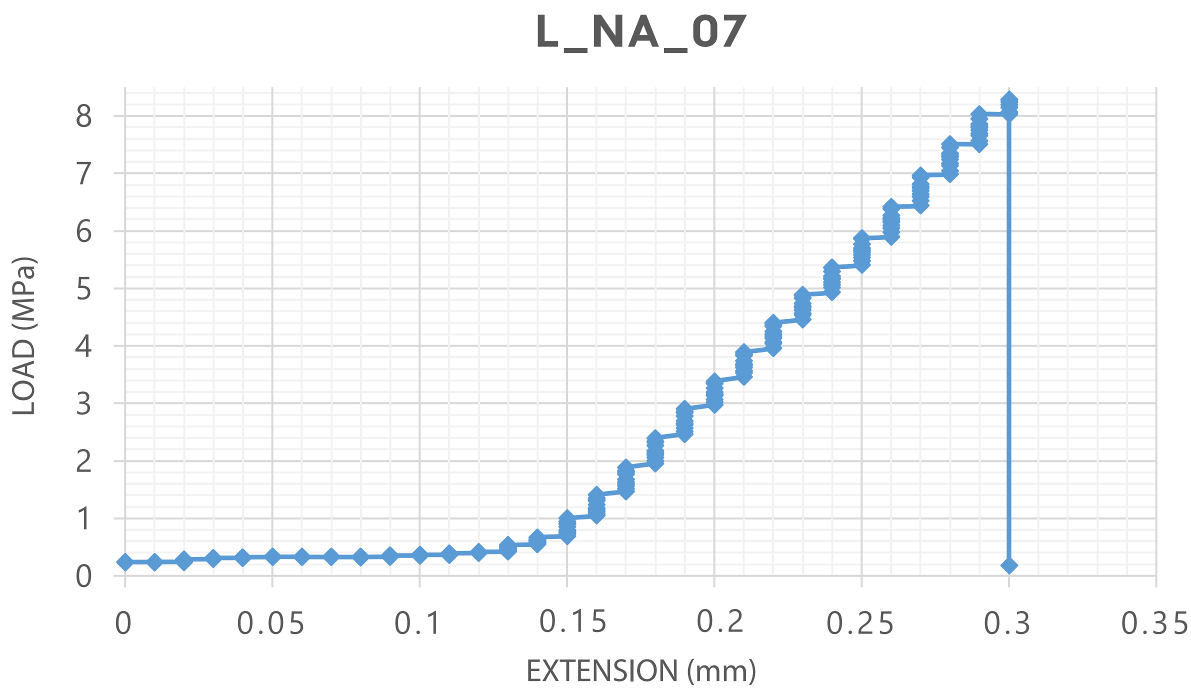
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_NA_06
LOAD (MPa)
8
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)



L_NA_07
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5
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0
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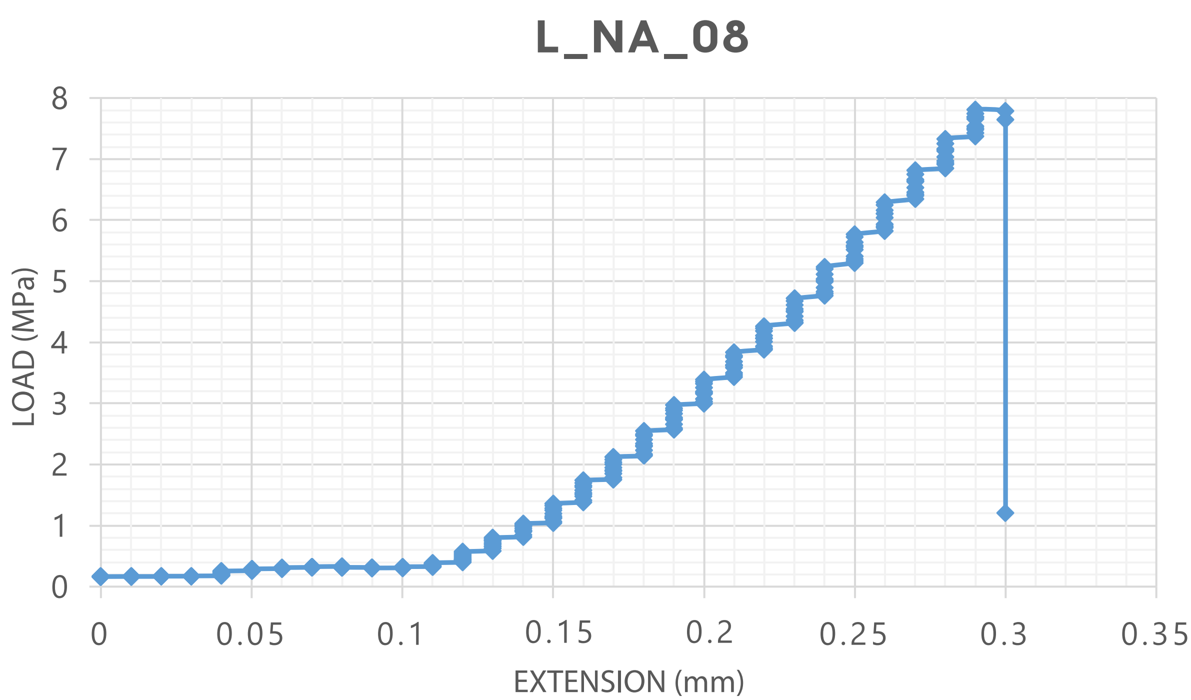
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)



L_NA_08
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5
4
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2
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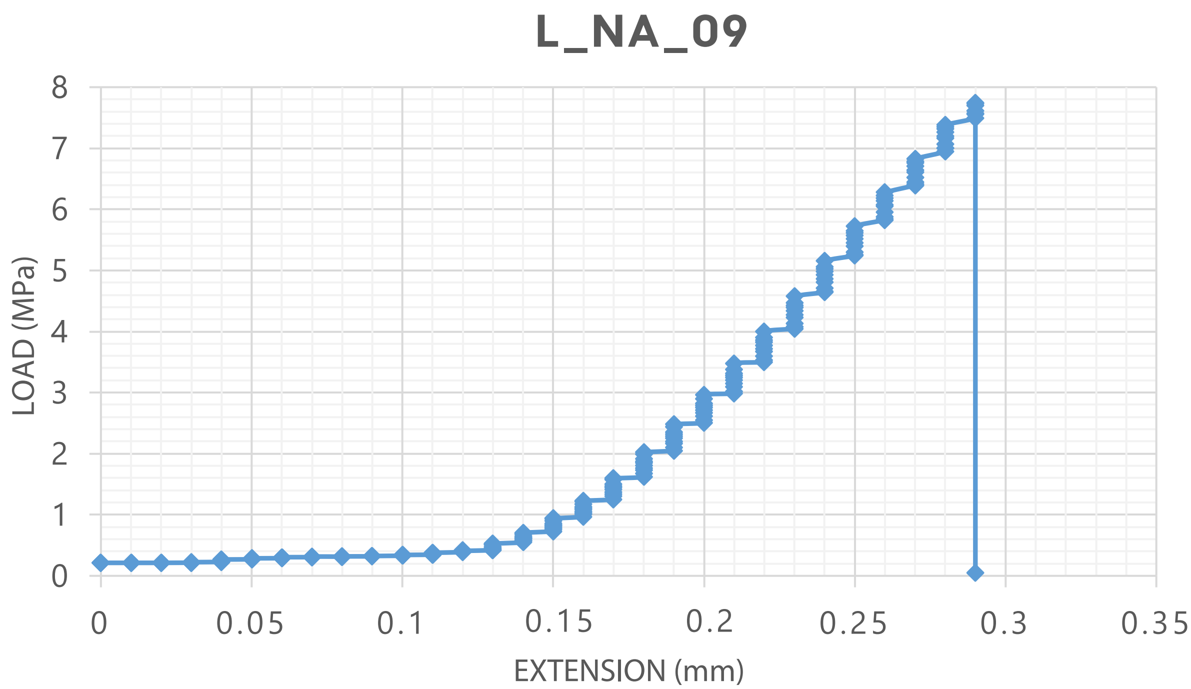
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_NA_09
LOAD (MPa)
EXTENSION (mm)
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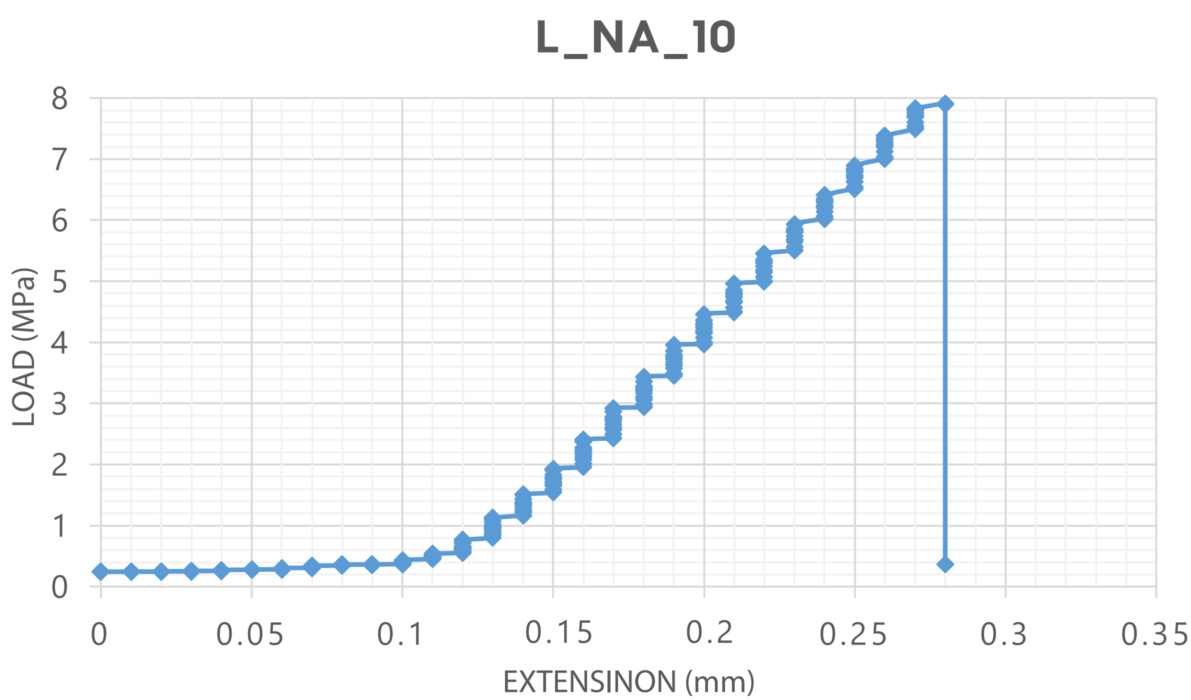
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_NA_10
LOAD (MPa)
EXTENSINON (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35

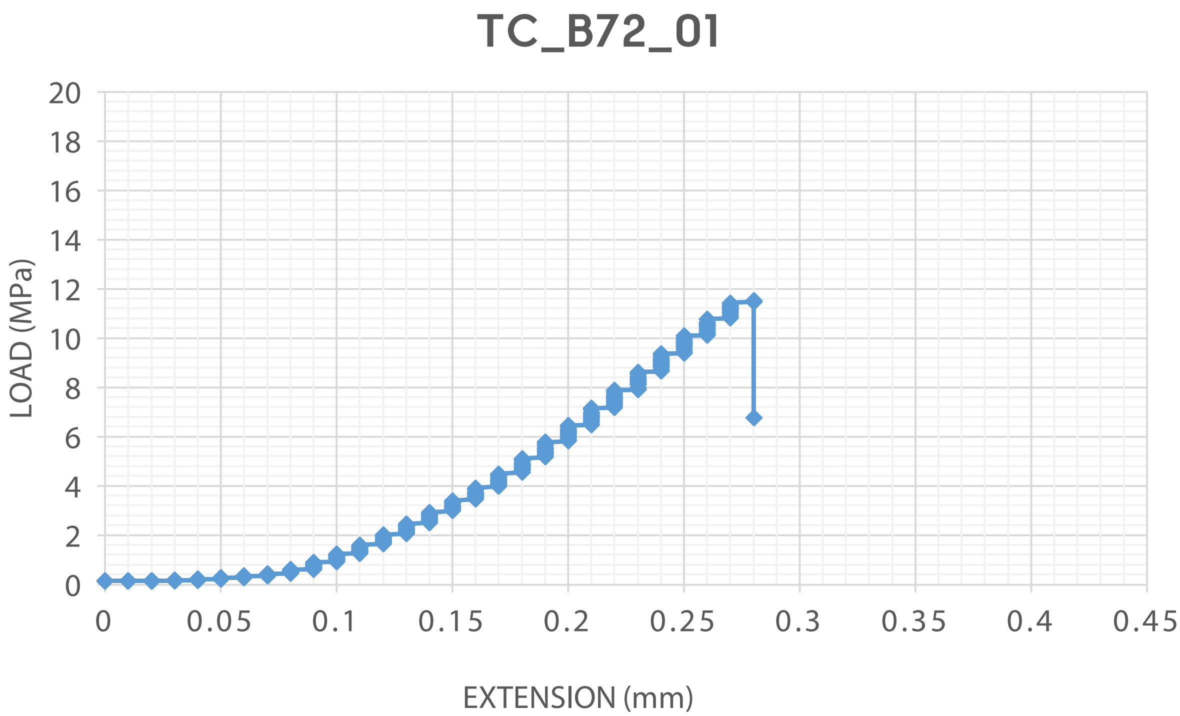
TERRA COTTA – GROUP A

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_B72_01	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-720.00	Broke far from joint	11.81			1550.30
TC_B72_02	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-566.82	Broke far from joint	9.30			1317.73
TC_B72_03	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-431.49	Broke at joint	7.08			1584.55
TC_B72_04	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-608.70	Broke far from joint	9.98			1836.76
TC_B72_05	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-512.30	Broke far from joint	8.40			1336.75
TC_B72_06	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-690.29	Broke far from joint	11.32			1722.87
TC_B72_07	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-509.95	Broke far from joint	8.36			1181.28
TC_B72_08	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-707.94	Broke far from joint	11.61			1852.71
TC_B72_09	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-885.64	Broke far from joint	14.52			1852.24
TC_B72_10	2/1/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-511.81	Broke far from joint	8.39			105.99
											10.08	2.23	
TC_B48N_01	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-664.99	Broke next to joint	10.91			1247.01
TC_B48N_02	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-560.74	Broke at joint	9.20			1340.93
TC_B48N_03	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-564.67	Broke next to joint	9.26			1089.90
TC_B48N_04	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-672.34	Broke far from joint	11.03			1499.22
TC_B48N_05	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-540.44	Broke far from joint	8.86			1229.48
TC_B48N_06	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-880.64	Broke next to joint	14.44			1476.50
TC_B48N_07	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-772.96	Broke far from joint	12.68			1813.77
TC_B48N_08	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-1027.74	Broke far from joint	16.85			2039.70

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_B48N_09	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-811.11	Broke next to joint	13.30			2185.79
TC_B48N_10	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-690.58	Broke next to joint	11.33			1606.06
											11.79	2.56	
TC_B44_01	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-719.51	Broke far from joint	11.80			1648.25
TC_B44_02	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-1052.25	Broke far from joint	17.26			1878.44
TC_B44_03	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-1175.82	Broke far from joint	19.28			1984.06
TC_B44_04	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-898.29	Broke next to joint	14.73			2063.16
TC_B44_05	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-579.97	Broke far from joint	9.51			1133.92
TC_B44_06	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; possible glue expansion	4/4/2017	-759.53	Broke far from joint	12.46			1567.35
TC_B44_07	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; possible glue expansion	4/4/2017	-676.56	Broke far from joint	11.10			2453.12
TC_B44_08	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; possible glue expansion	4/4/2017	-613.20	Broke far from joint	10.06			1340.93
TC_B44_09	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; possible glue expansion	4/4/2017	-499.0	Broke at joint	8.18			1328.04
TC_B44_10	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; possible glue expansion	4/4/2017	-1006.15	No visible failure	16.50			2006.44
											13.09	3.68	
TC_A11_01	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-726.38	Broke far from joint	11.91			1542.67
TC_A11_02	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-716.08	Broke at joint	11.74			1380.98
TC_A11_03	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-644.20	Broke far from joint	10.56			1485.00
TC_A11_04	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-613.11	Broke at joint	10.06			1424.99
TC_A11_05	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-754.03	Broke at joint	12.37			1931.85
TC_A11_06	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-573.69	Broke far from joint	9.41			1121.04

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_A11_07	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-866.72	Broke at joint	14.21			1925.61
TC_A11_08	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-748.25	Broke far from joint	12.27			1414.34
TC_A11_09	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-841.41	Broke far from joint	13.80			1527.62
TC_A11_10	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-468.37	Broke far from joint	7.68			1307.00
											11.40	2.00	
TC_1B72_3B48N_01	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-771.10	Broke at joint	12.65			2305.32
TC_1B72_3B48N_02	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-508.08	Broke at joint	8.33			1751.29
TC_1B72_3B48N_03	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-712.65	Broke at joint	11.69			2134.76
TC_1B72_3B48N_04	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-875.64	Broke far from joint	14.36			3844.83
TC_1B72_3B48N_05	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-769.04	Broke at joint	12.61			1594.29
TC_1B72_3B48N_06	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-866.42	Broke far from joint	14.21			1680.93
TC_1B72_3B48N_07	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-884.85	Broke at joint	14.51			1304.22
TC_1B72_3B48N_08	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-548.09	Broke at joint	8.99			1024.51
TC_1B72_3B48N_09	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-906.72	Broke far from joint	14.87			1618.80
TC_1B72_3B48N_10	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-608.11	Broke far from joint	9.97			1291.57
											12.22	2.40	
TC_3B72_1B48N_01	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-646.55	Broke far from joint	10.60			1291.75
TC_3B72_1B48N_02	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-525.73	Broke far from joint	8.62			1348.43
TC_3B72_1B48N_03	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-517.59	Broke far from joint	8.49			932.31
TC_3B72_1B48N_04	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-593.11	Broke far from joint	9.73			1199.50

	Bonding Start Date	Bonding End Date	Roll Test 1 3/13/17	Thermal Cycling Start Date	Thermal Cycling End Date	Roll Test 2	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_3B72_1B48N_05	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-1017.93	Broke far from joint	16.69			2123.90
TC_3B72_1B48N_06	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-909.86	Broke far from joint	14.92			2108.75
TC_3B72_1B48N_07	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-510.24	Broke far from joint	8.37			1212.57
TC_3B72_1B48N_08	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-684.50	Broke at joint	11.23			1124.67
TC_3B72_1B48N_09	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-569.96	Broke far from joint	9.35			1322.12
TC_3B72_1B48N_10	2/2/2017	3/13/2017	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled; but stuck to the metal grate	4/4/2017	-539.66	Broke far from joint	8.85			1423.74
											10.68	2.88	
TC_CT_NA_01	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-817.97		13.41			14500.48
TC_CT_NA_02	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-929.08		15.24			1219.96
TC_CT_NA_03	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-906.43		14.87			1520.59
TC_CT_NA_04	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.				N/A			N/A
TC_CT_NA_05	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-516.03		8.46			1464.97
TC_CT_NA_06	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-565.75		9.28			1417.45
TC_CT_NA_07	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-404.52		6.63			882.18
TC_CT_NA_08	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-286.65		4.70			710.09
TC_CT_NA_09	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-720.59		11.82			1607.63
TC_CT_NA_10	N/A	N/A	Rolled on a flat surface.	3/17/2017	4/3/2017	Rolled on a flat surface.	4/4/2017	-759.53		12.46			10039.95
											10.76	3.69	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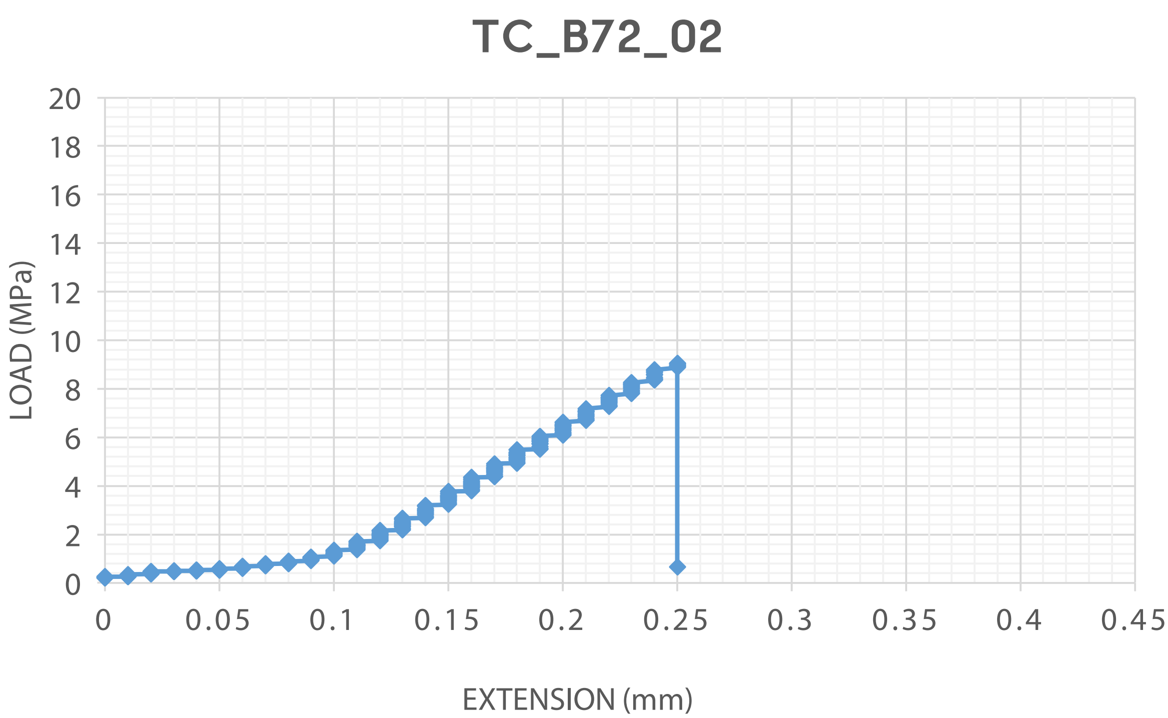
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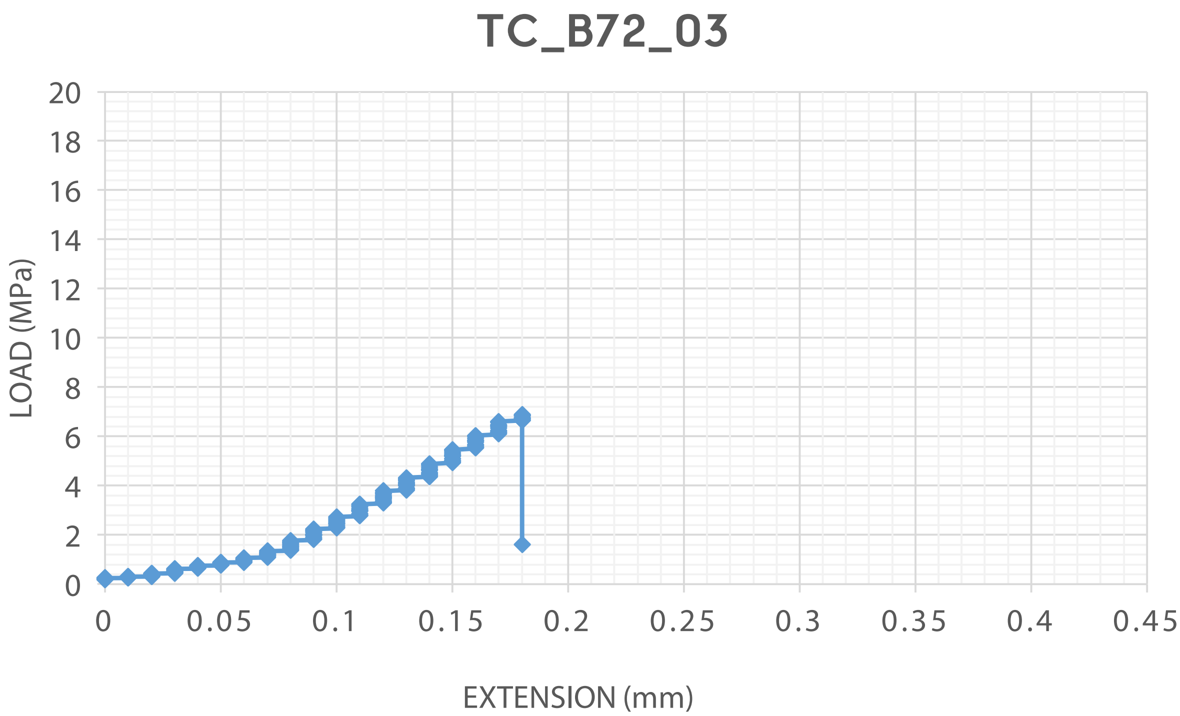

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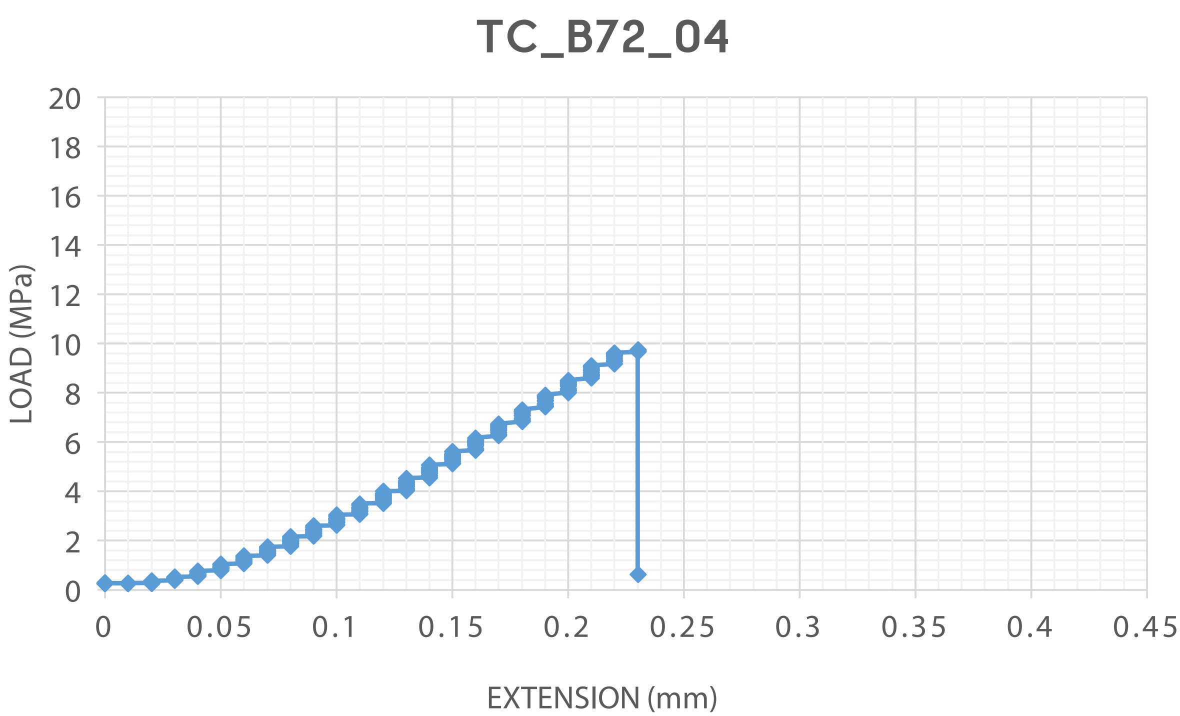

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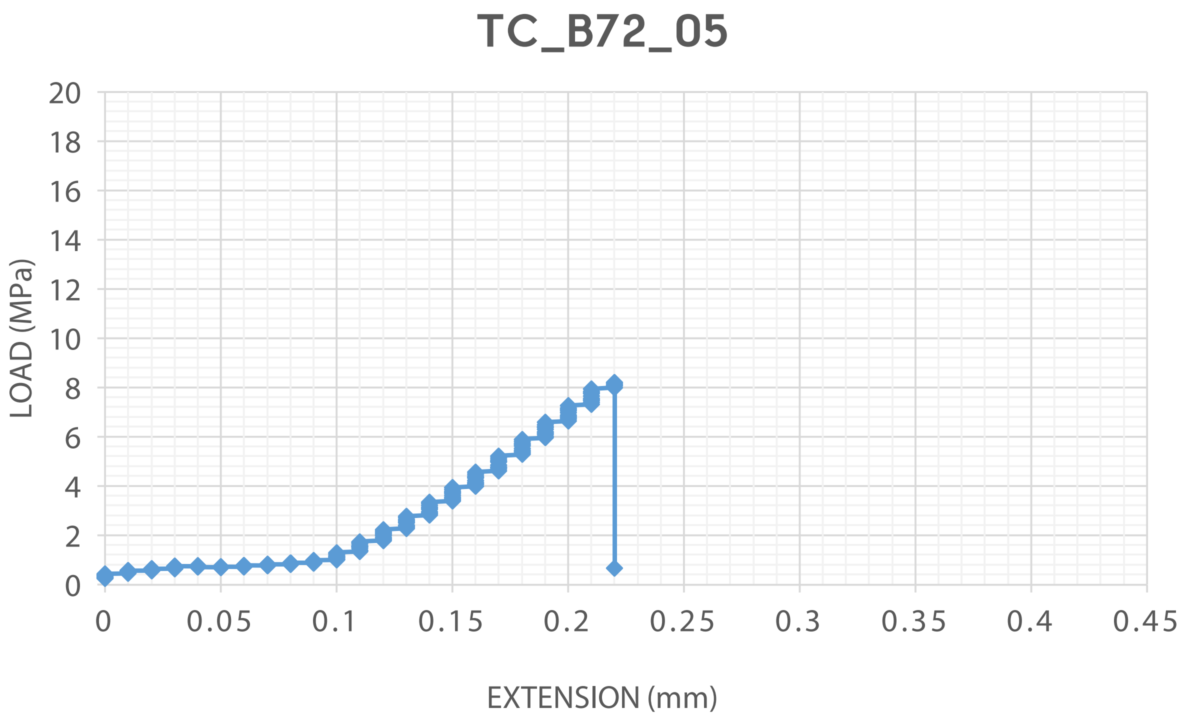
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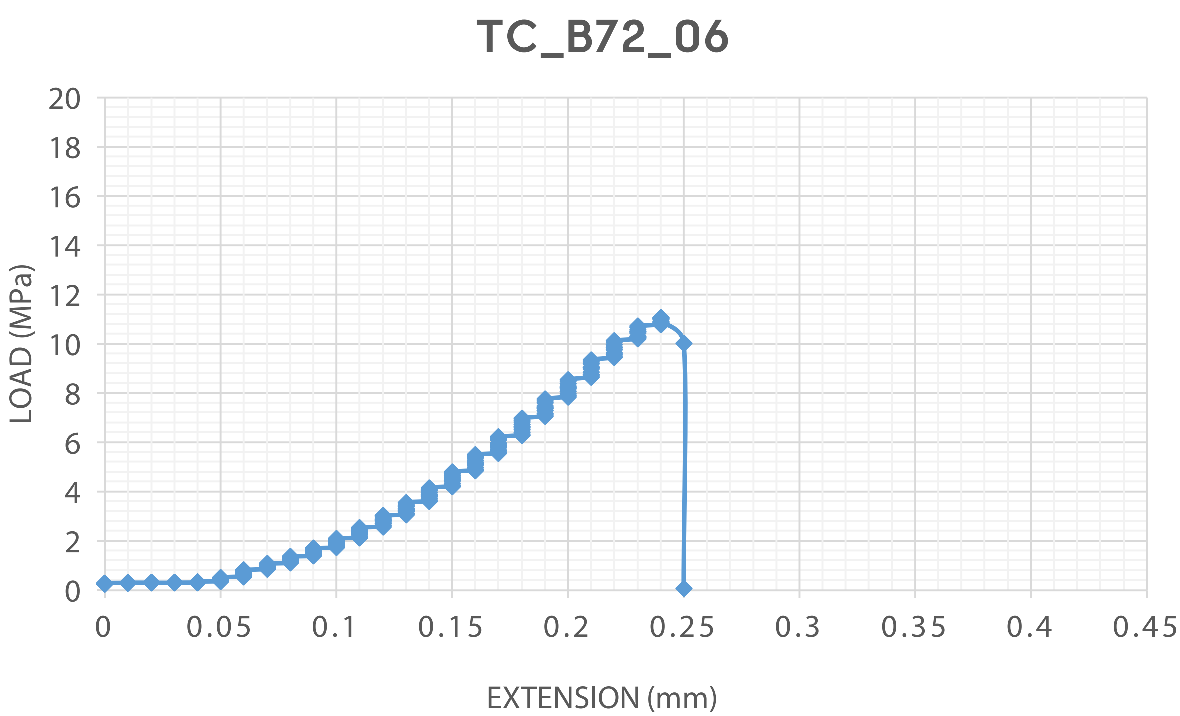
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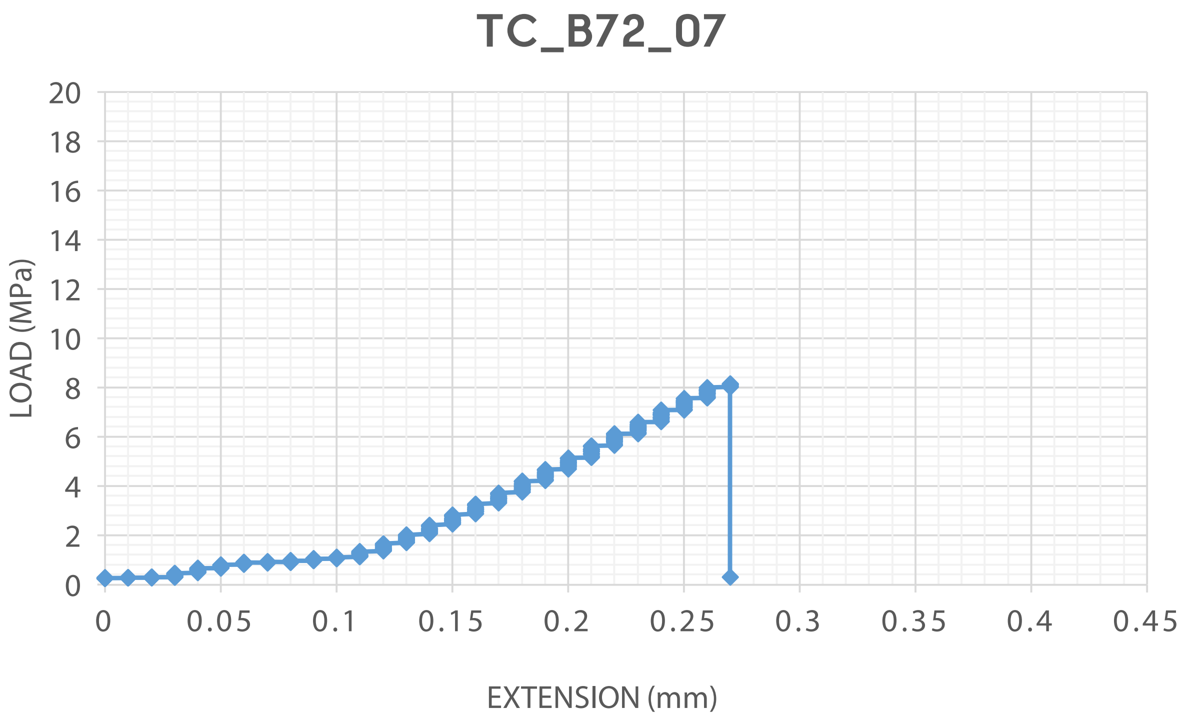
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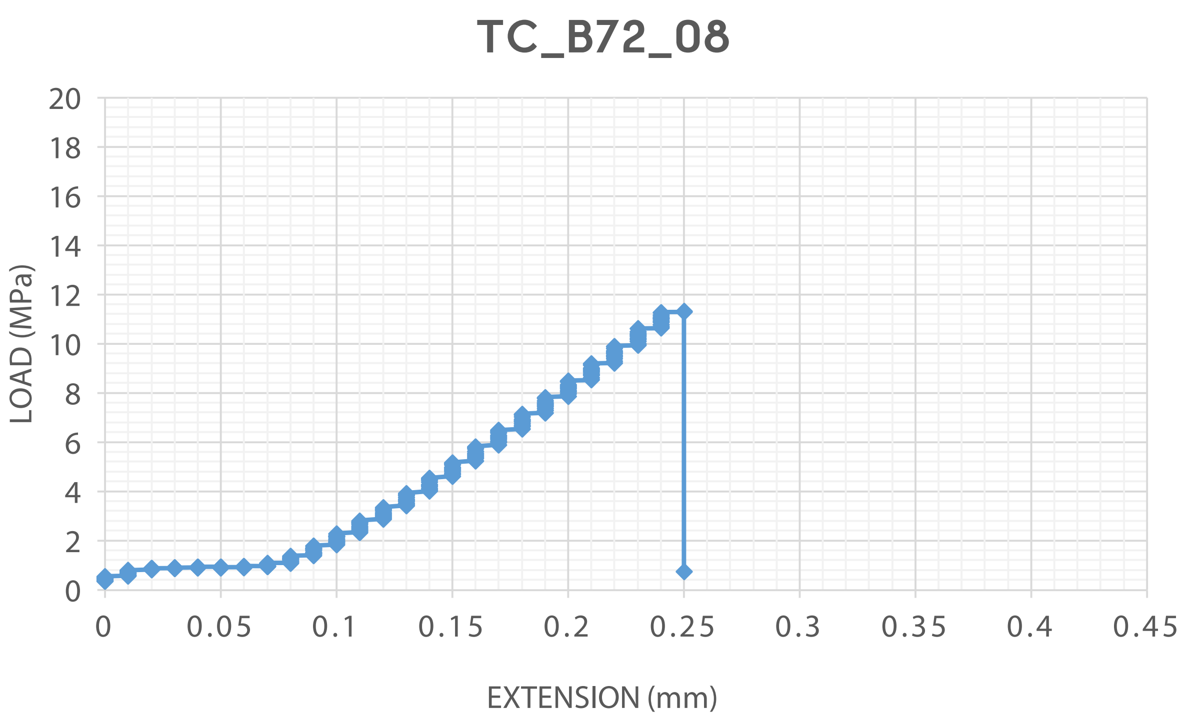
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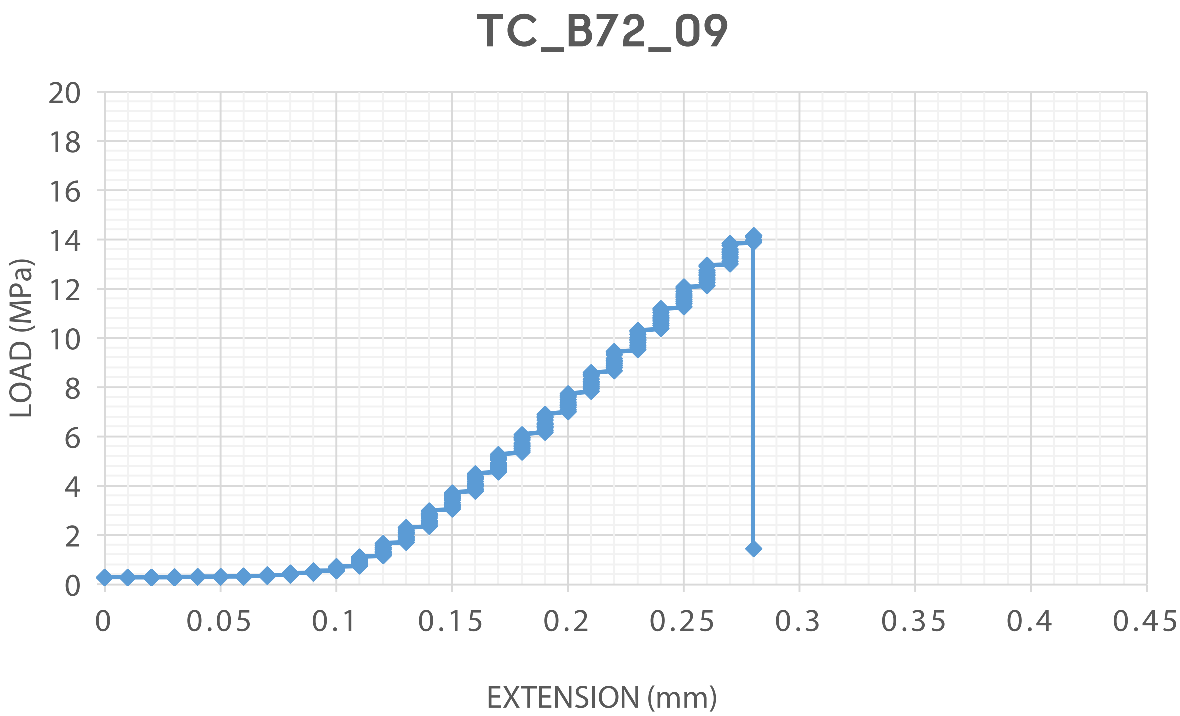
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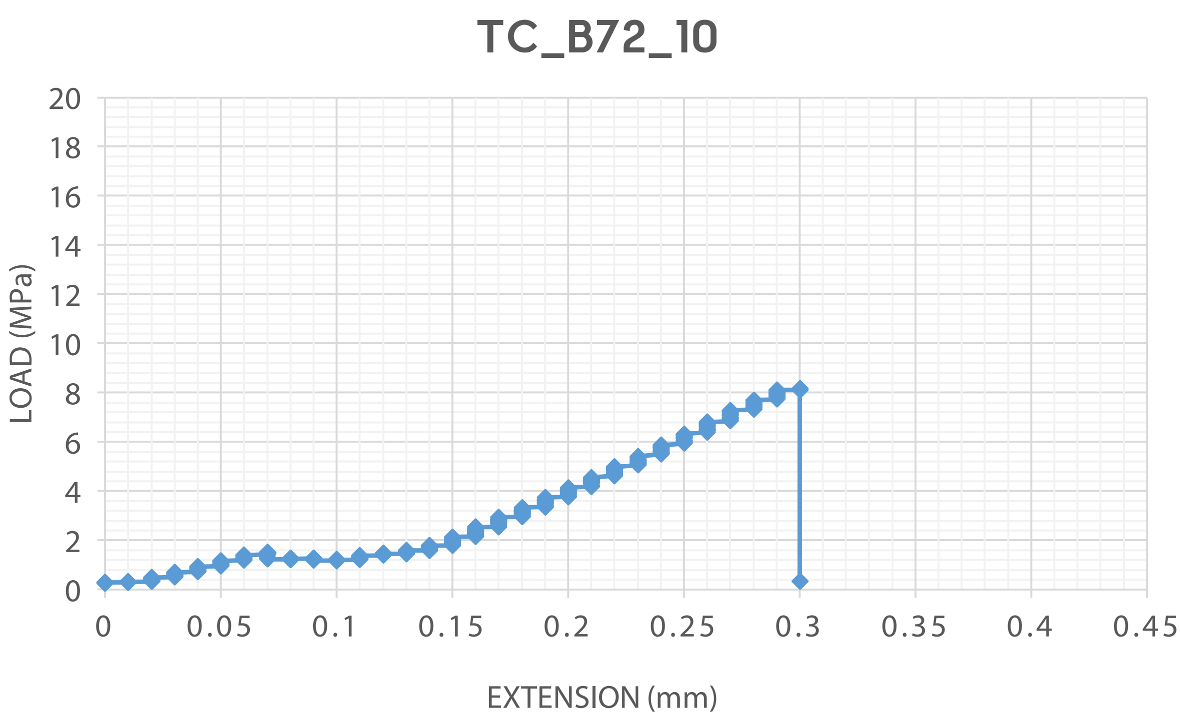
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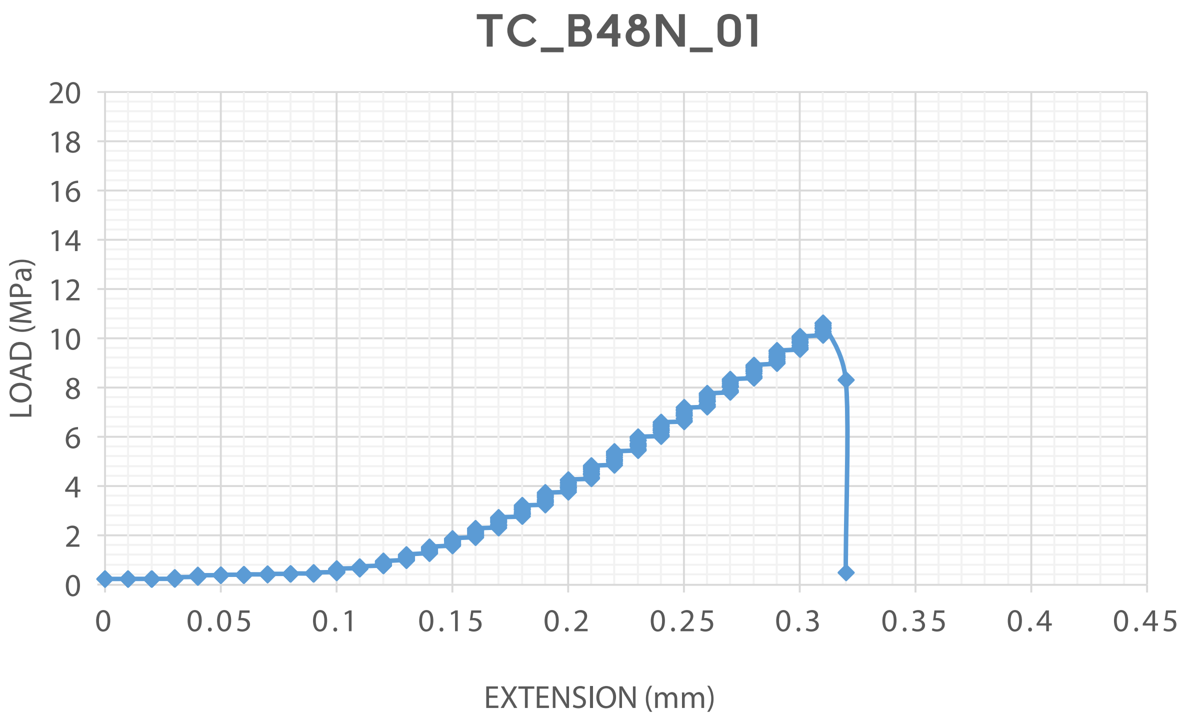
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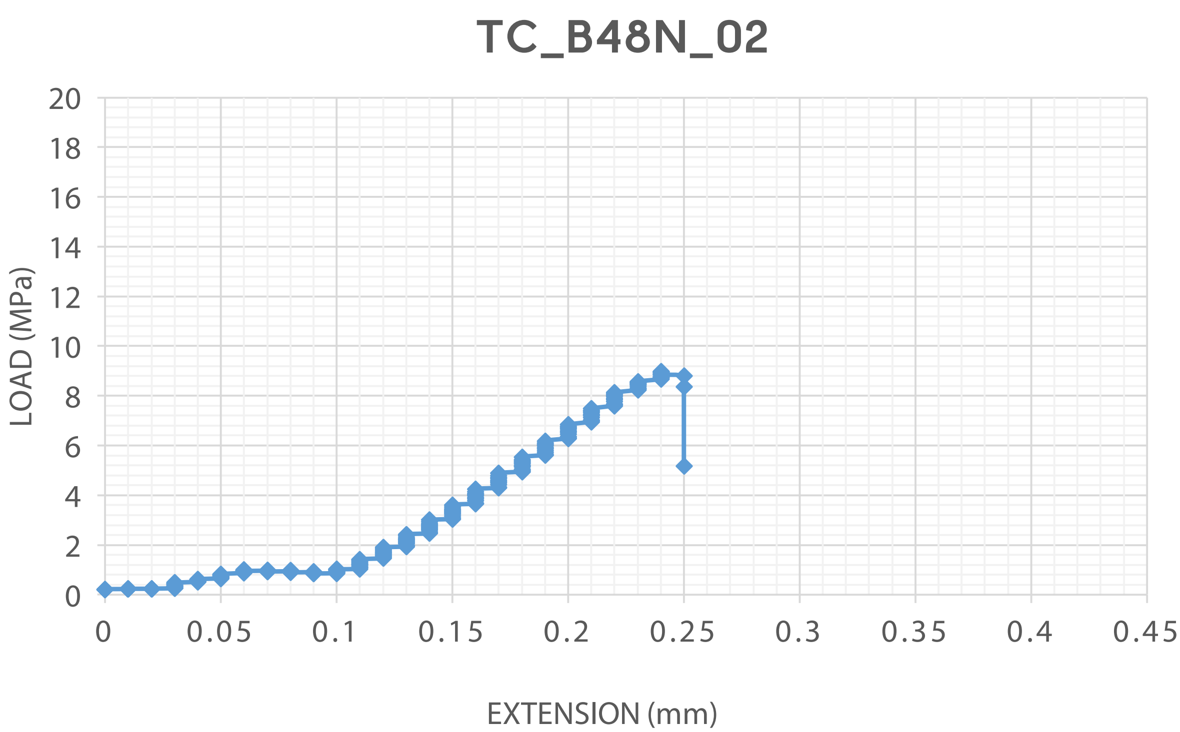
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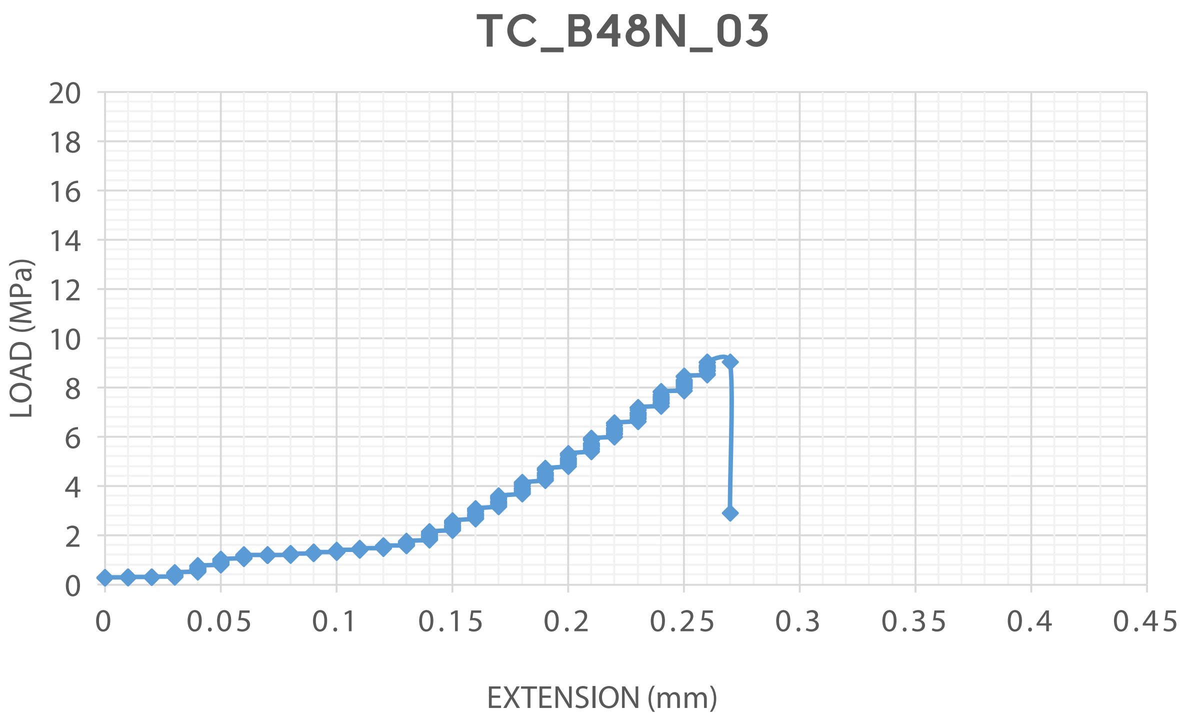
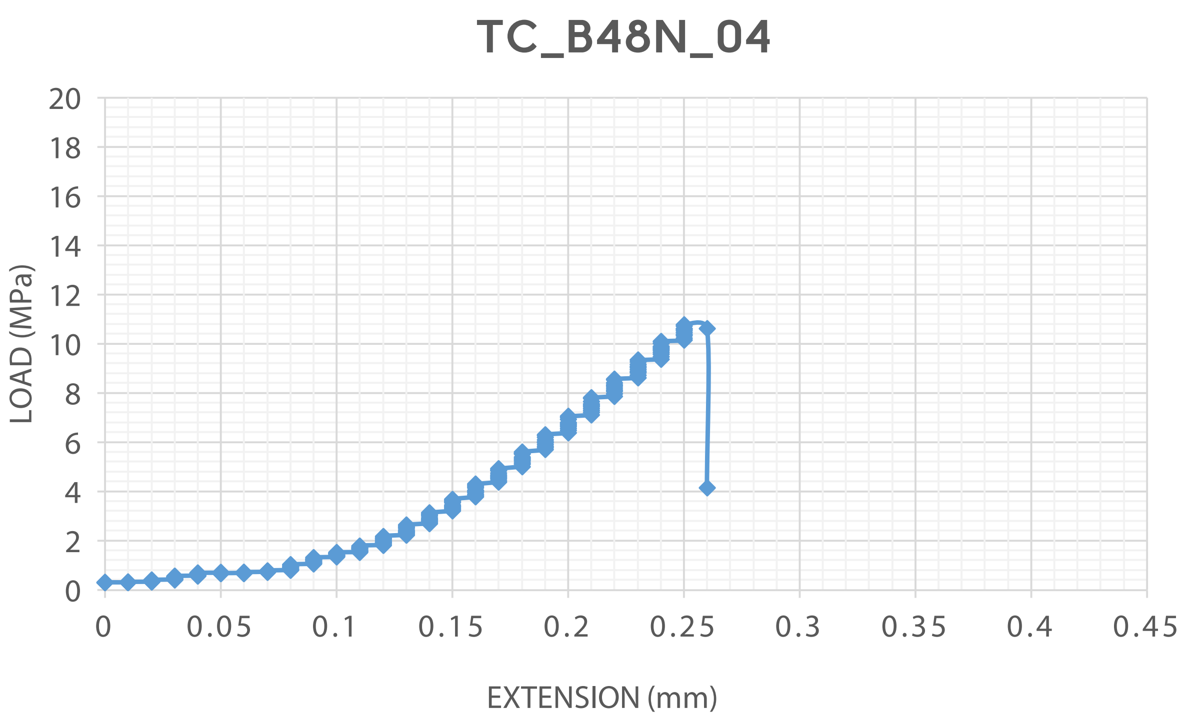
0.45
EXTENSION (mm)

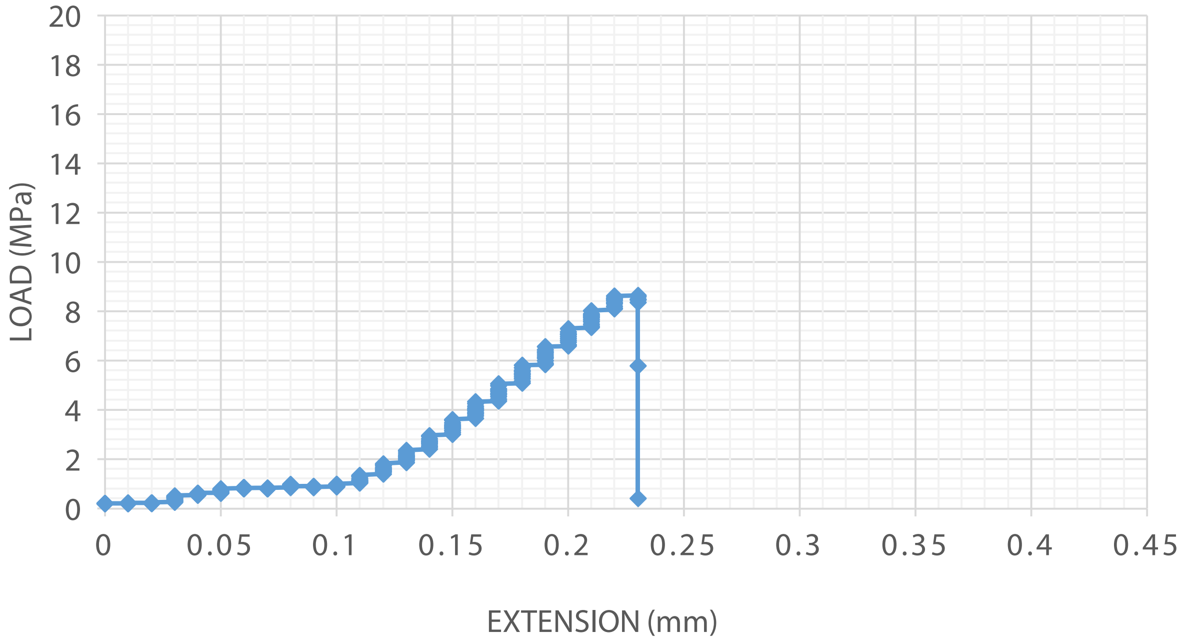
TC_B48N_03



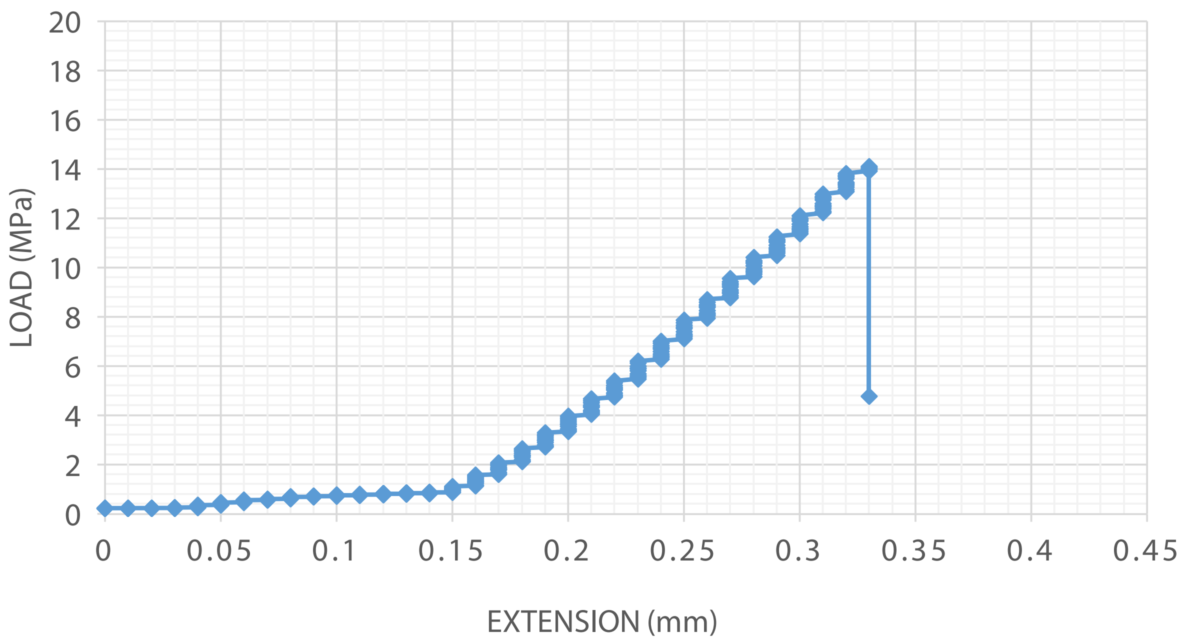
TC_B48N_04



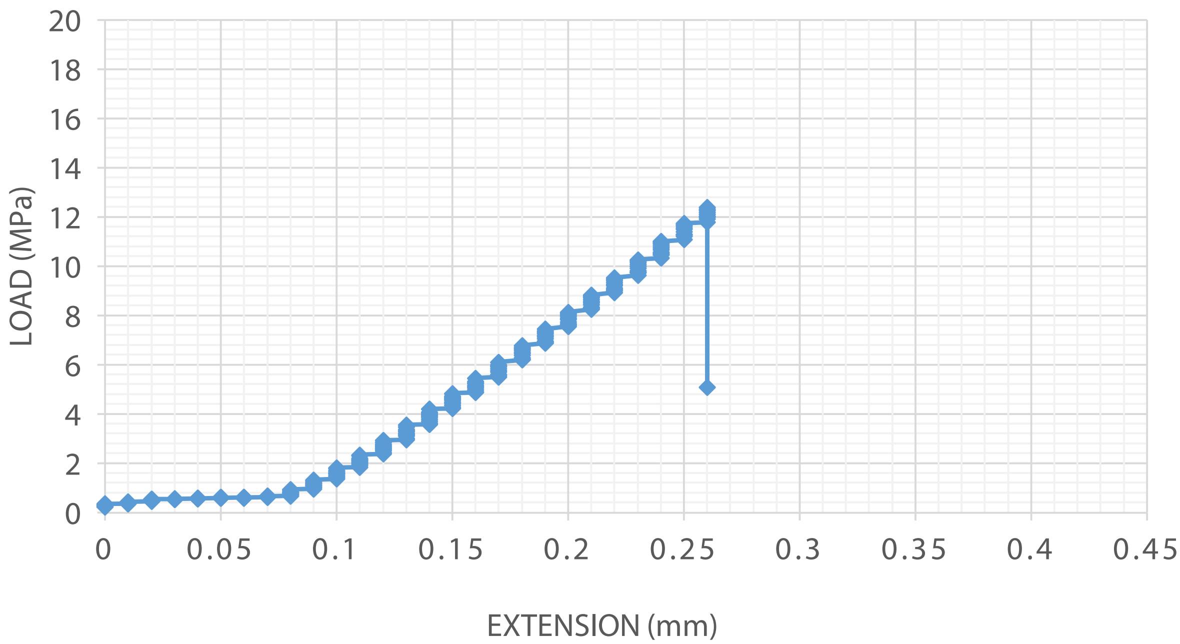
TC_B48N_05



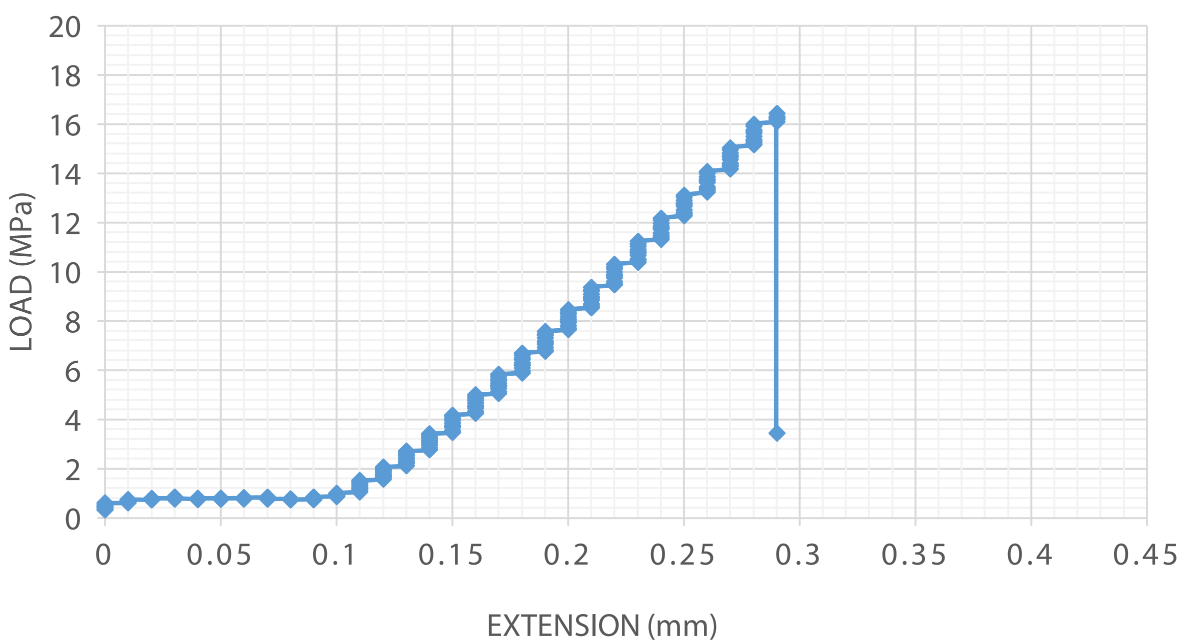
TC_B48N_06

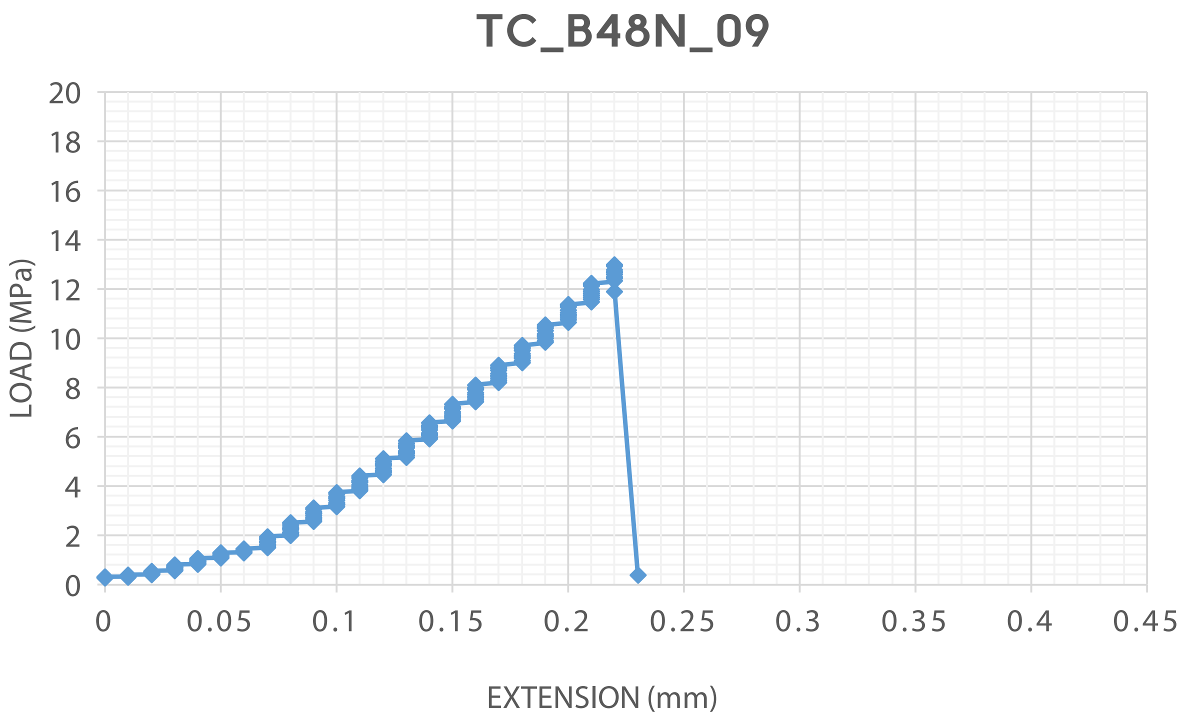


TC_B48N_07



TC_B48N_08




TC_B48N_09
LOAD (MPa)
EXTENSION (mm)
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8
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0
0.05
0.1
0.15
0.2
0.25
0.3
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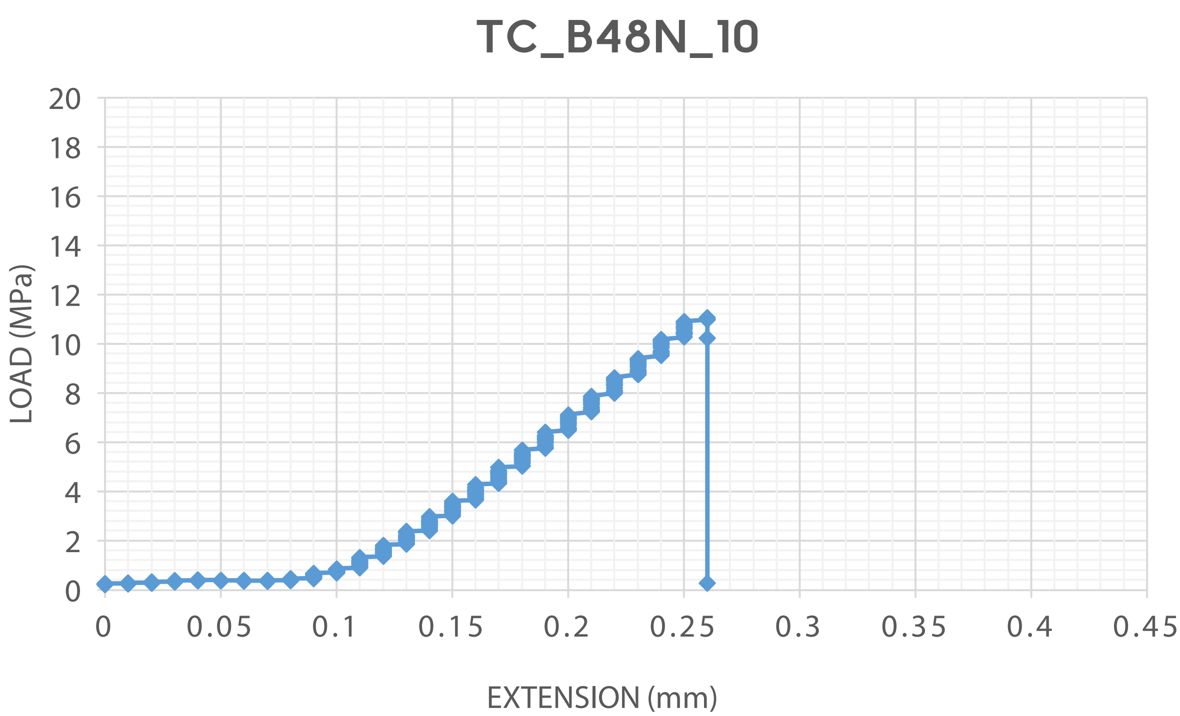
0.4
0.45


TC_B48N_10
LOAD (MPa)
EXTENSION (mm)
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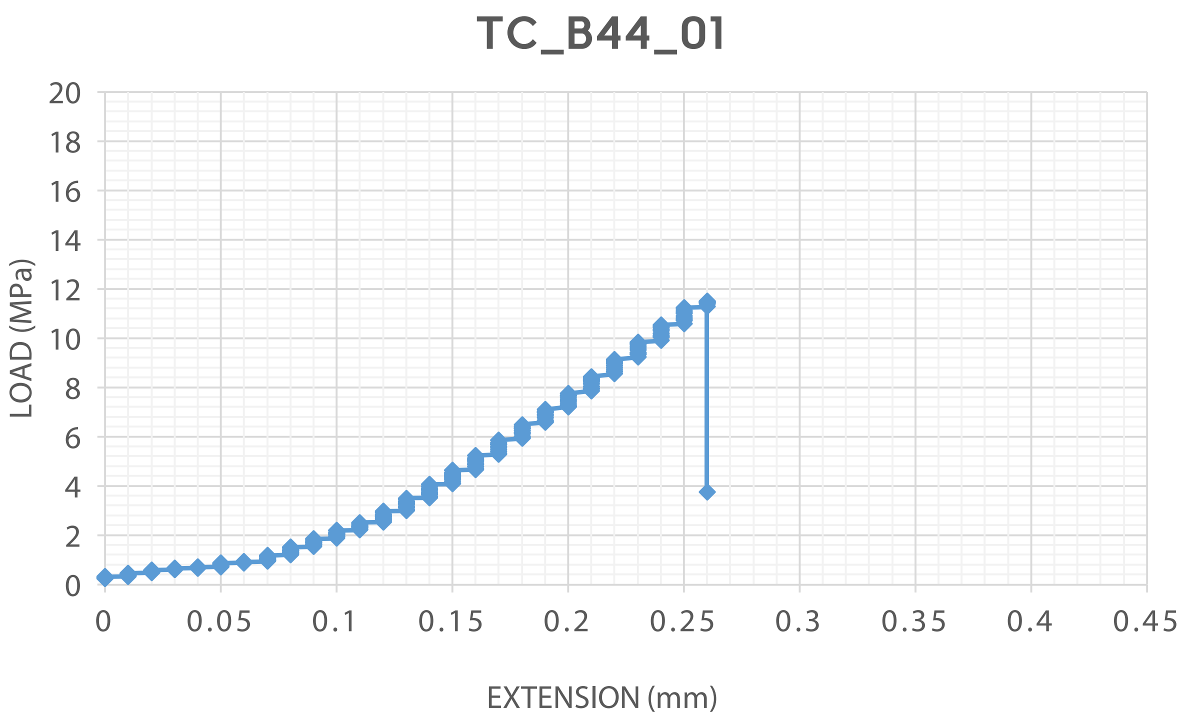
0.2
0.25
0.3
0.35
0.4
0.45


TC_B44_01
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EXTENSION (mm)
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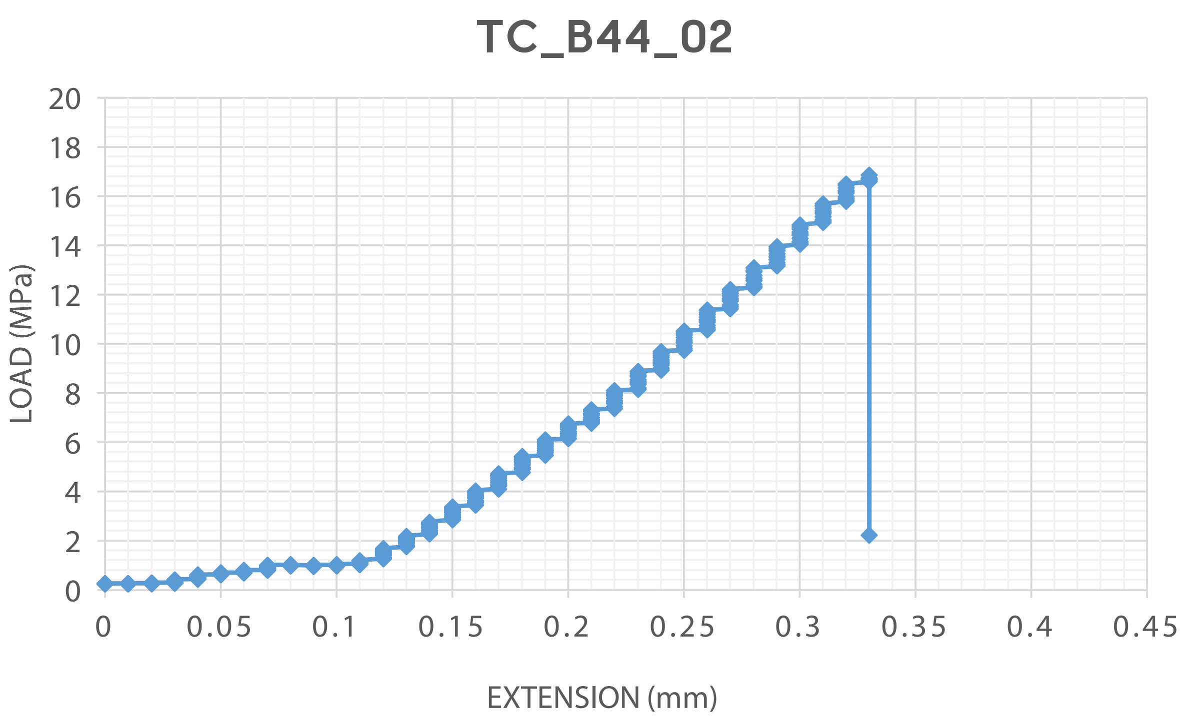
0.35
0.4
0.45


TC_B44_02
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EXTENSION (mm)
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0.05
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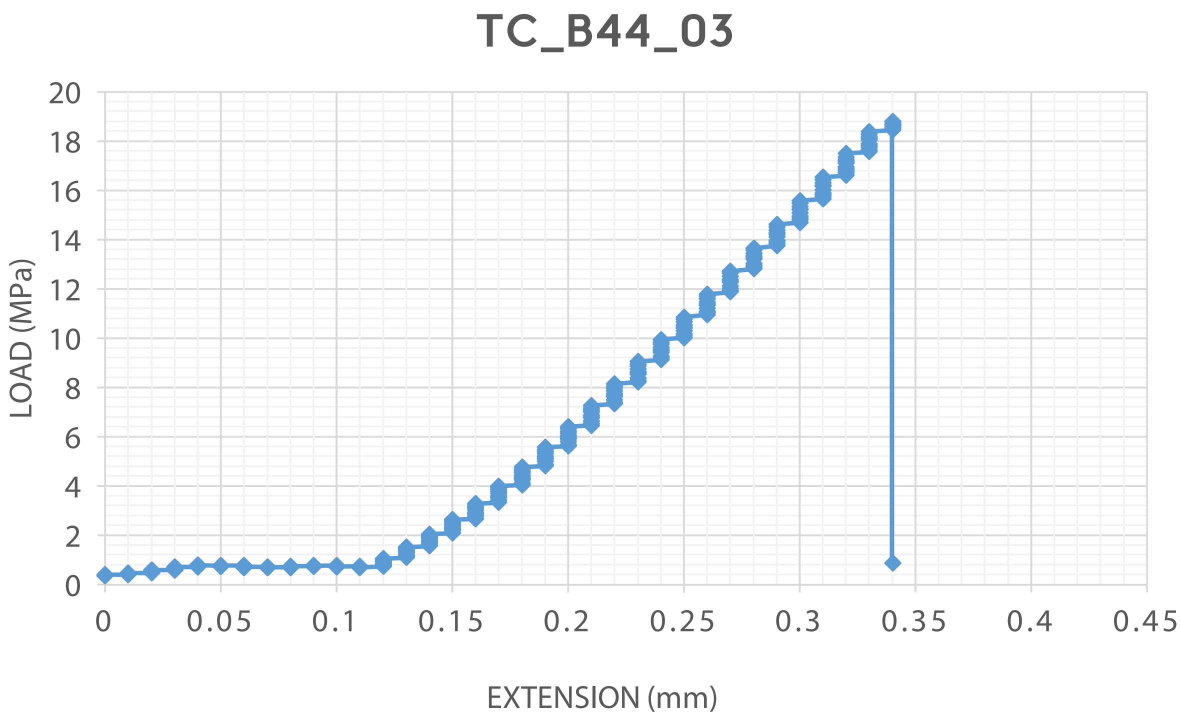
0.25
0.3
0.35
0.4
0.45



TC_B44_03
LOAD (MPa)
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14
12
10
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6
4
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0.1
0.15
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0.25
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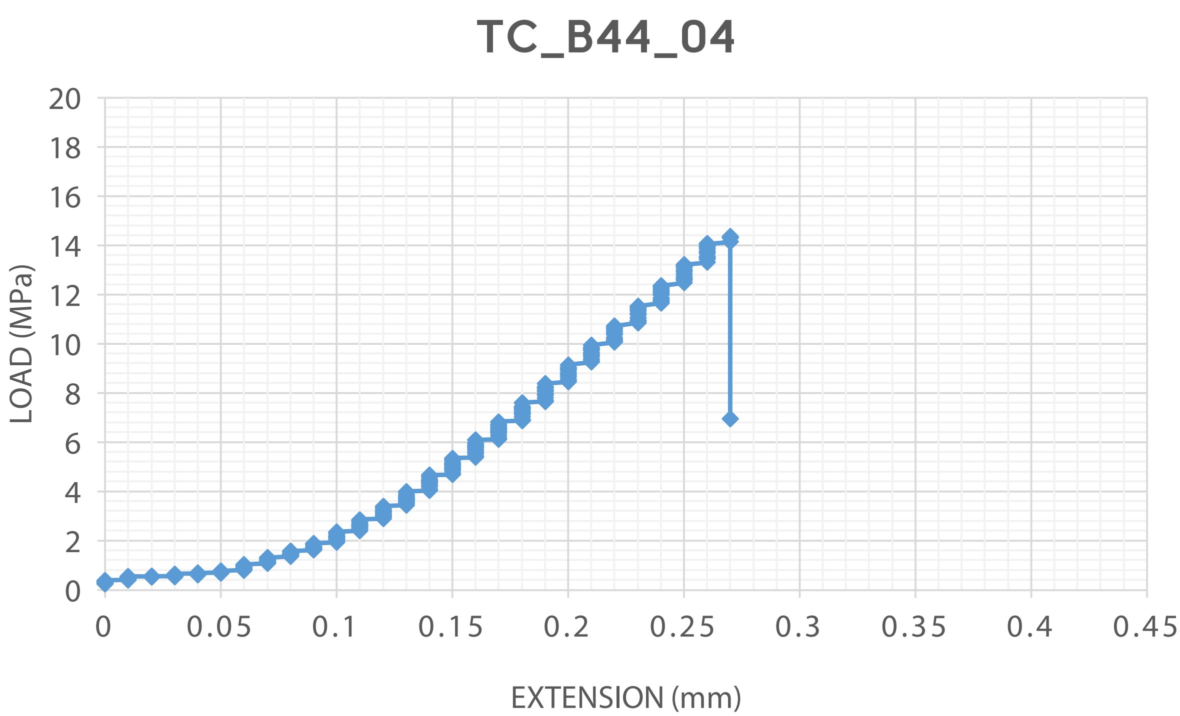
0.35
0.4
0.45
EXTENSION (mm)



TC_B44_04
LOAD (MPa)
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0.15
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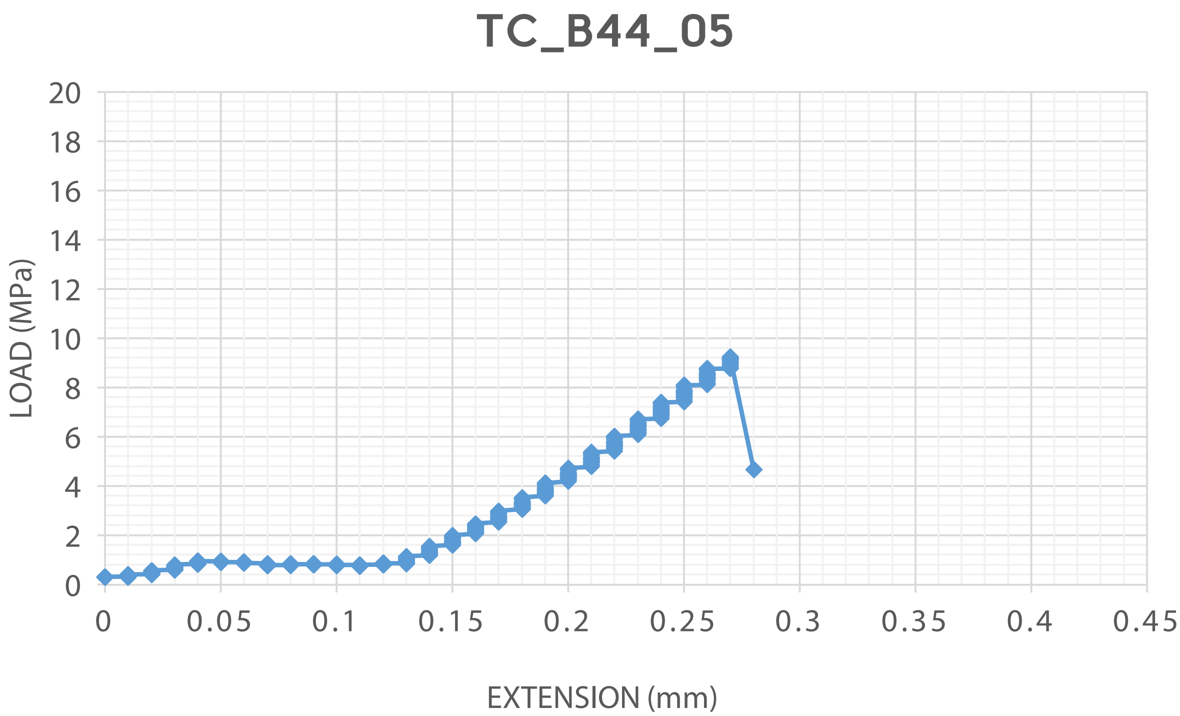
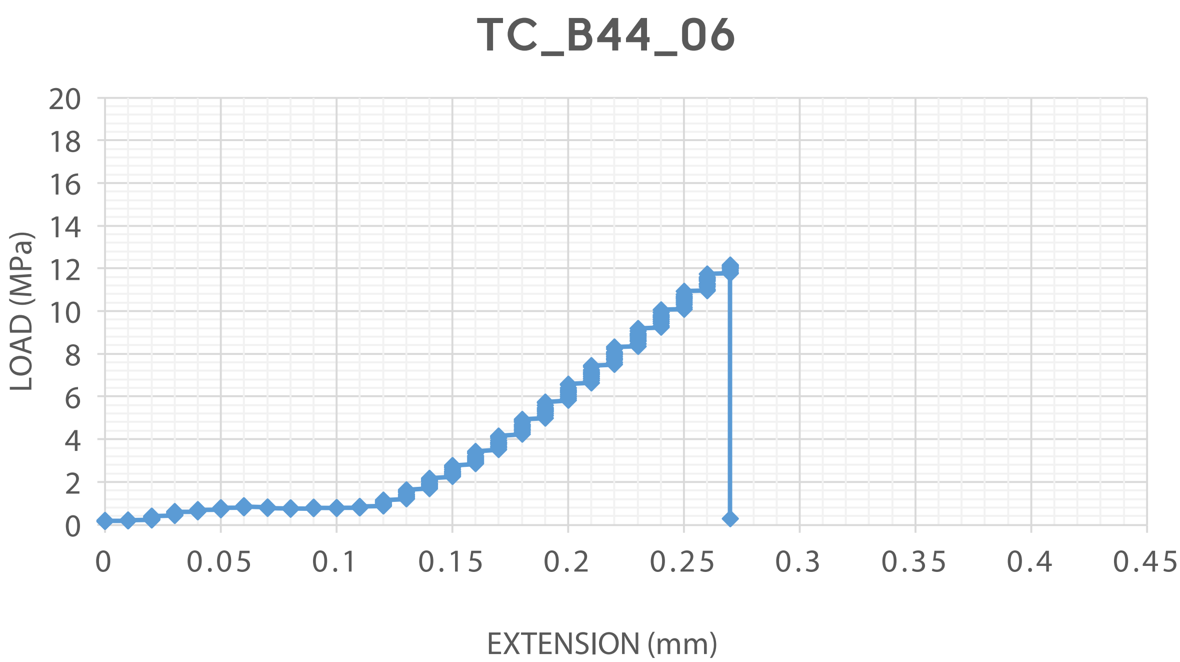
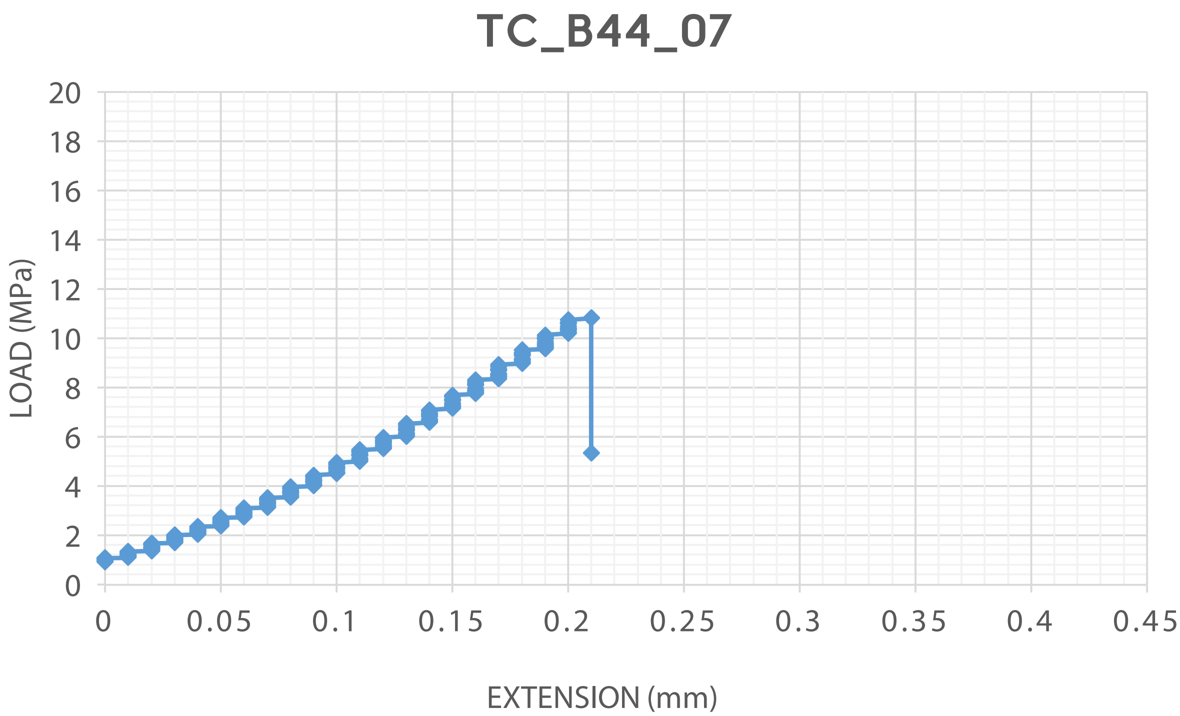
0.35
0.4
0.45
EXTENSION (mm)



TC_B44_05
LOAD (MPa)
EXTENSION (mm)



TC_B44_06
LOAD (MPa)
EXTENSION (mm)



TC_B44_07
LOAD (MPa)
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0.1
0.15
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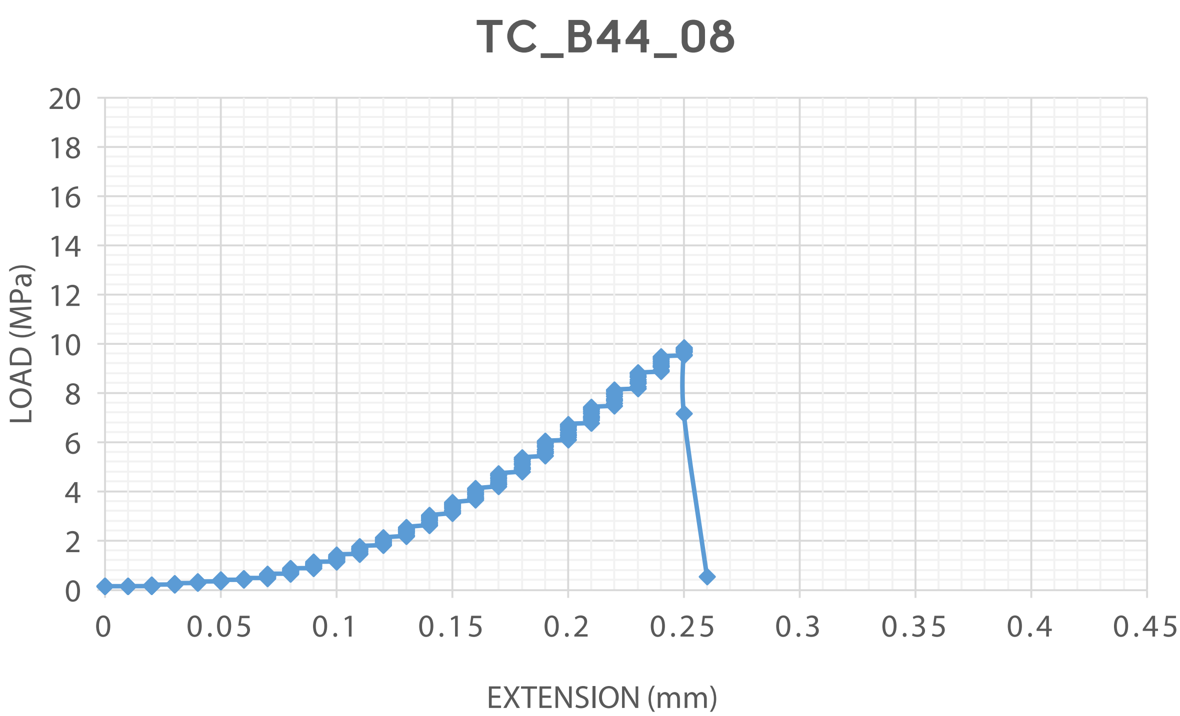
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)



TC_B44_08
LOAD (MPa)
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8
6
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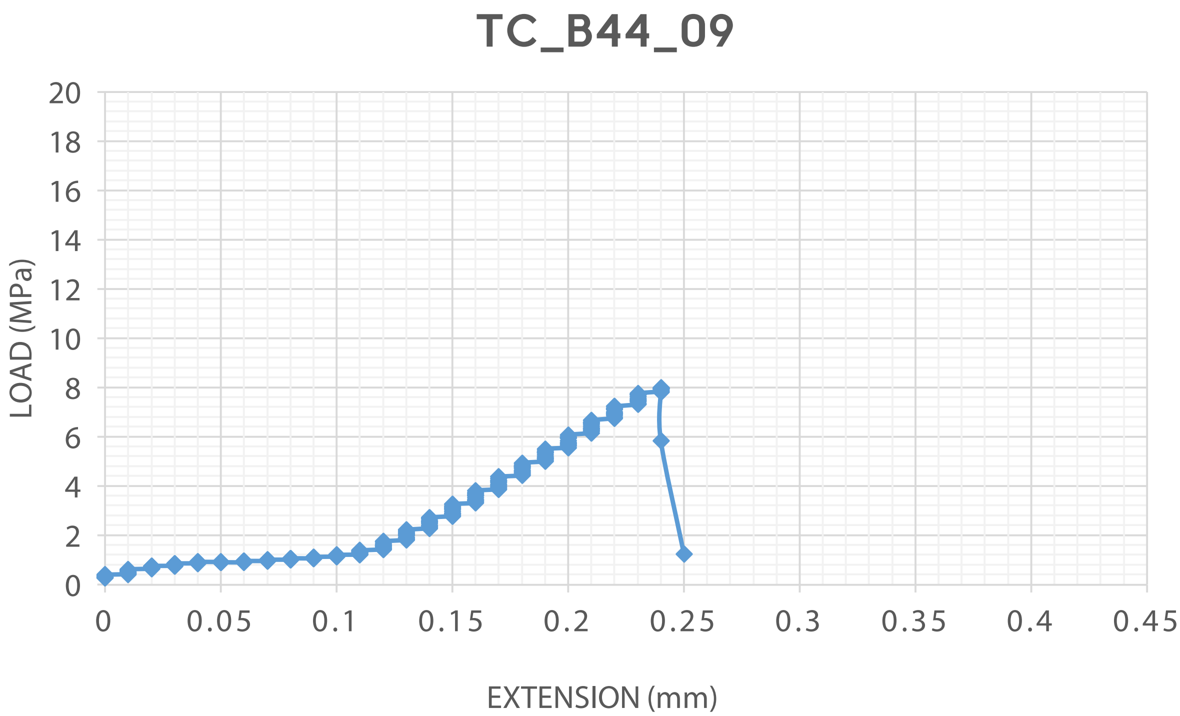
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_B44_09
LOAD (MPa)
EXTENSION (mm)
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2
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10
12
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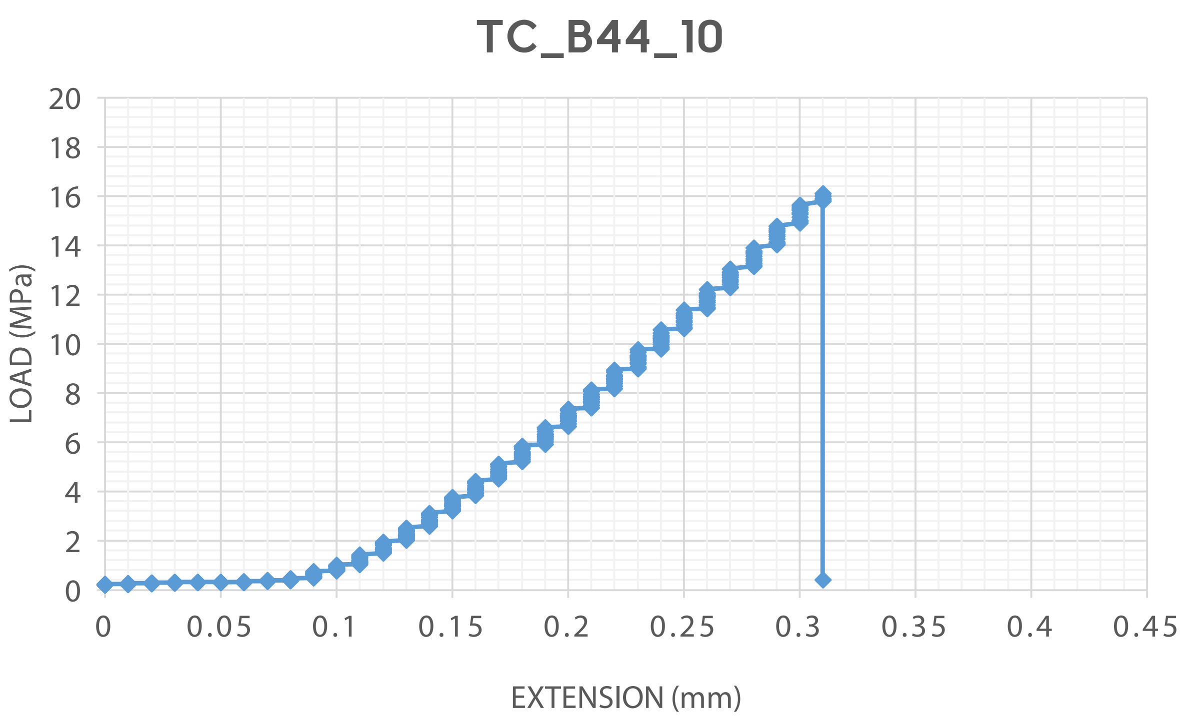
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_B44_10
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EXTENSION (mm)
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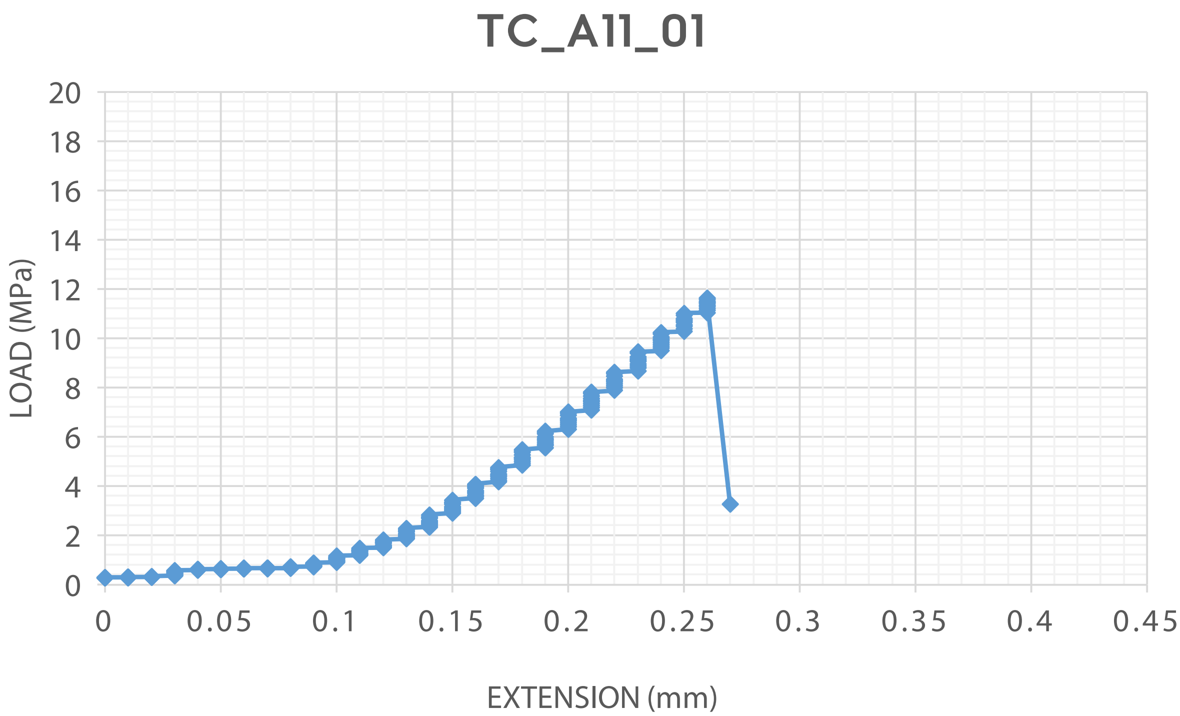
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_A11_01
LOAD (MPa)
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6
4
2
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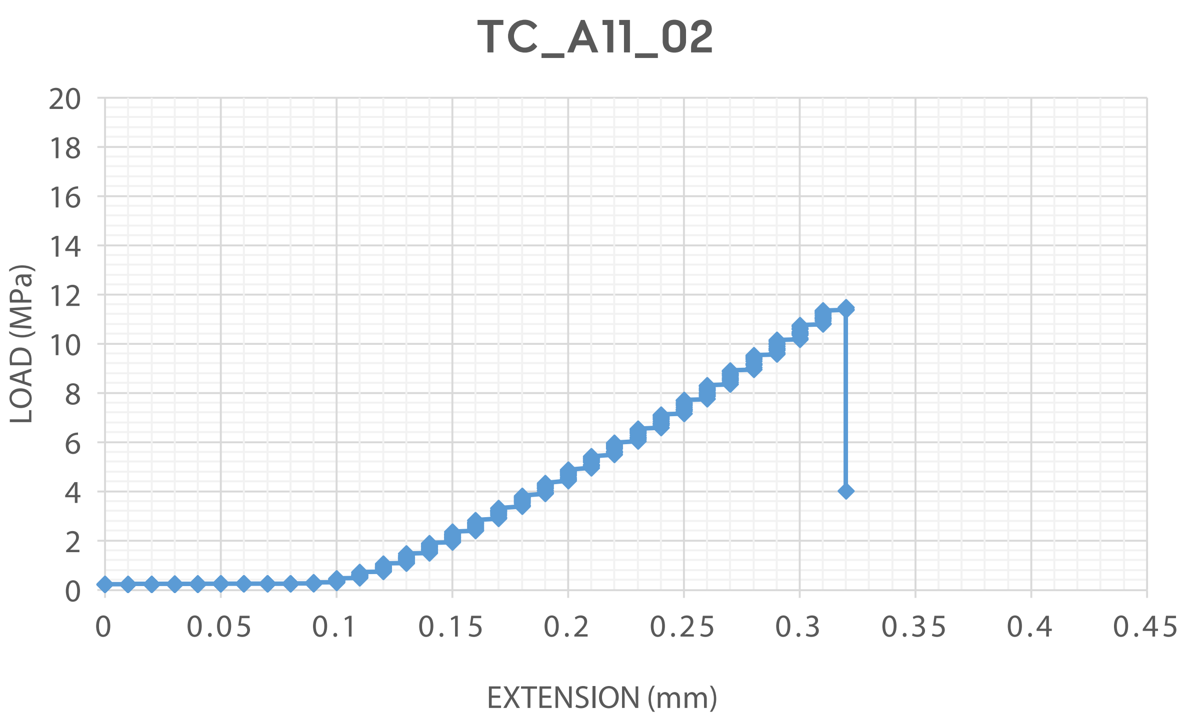
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_A11_02
LOAD (MPa)
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6
4
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0.1
0.15
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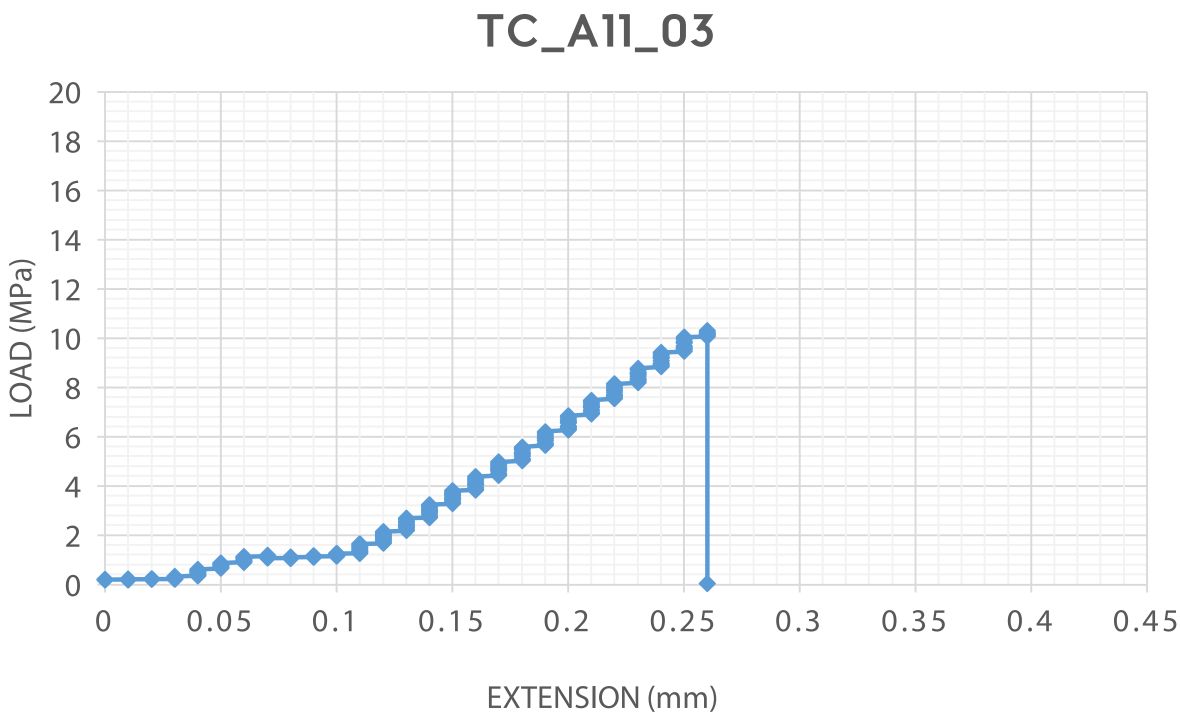
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)



TC_A11_03
LOAD (MPa)
EXTENSION (mm)
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0.15
0.2
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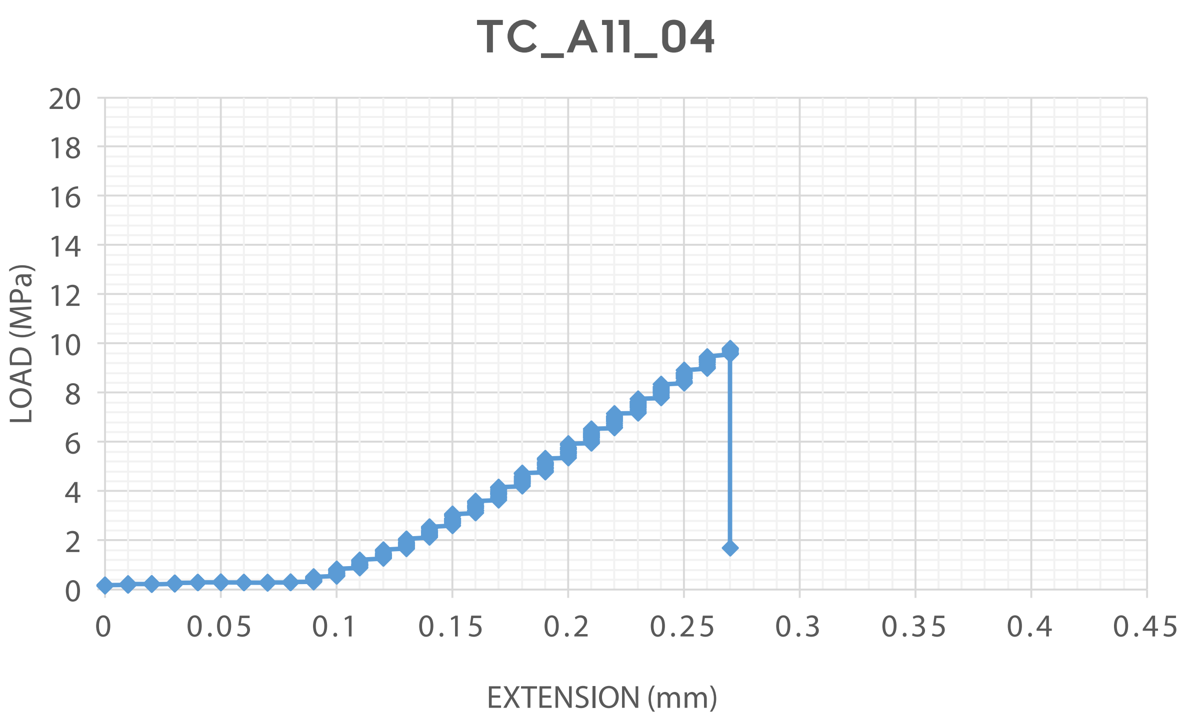
0.35
0.4
0.45



TC_A11_04
LOAD (MPa)
EXTENSION (mm)
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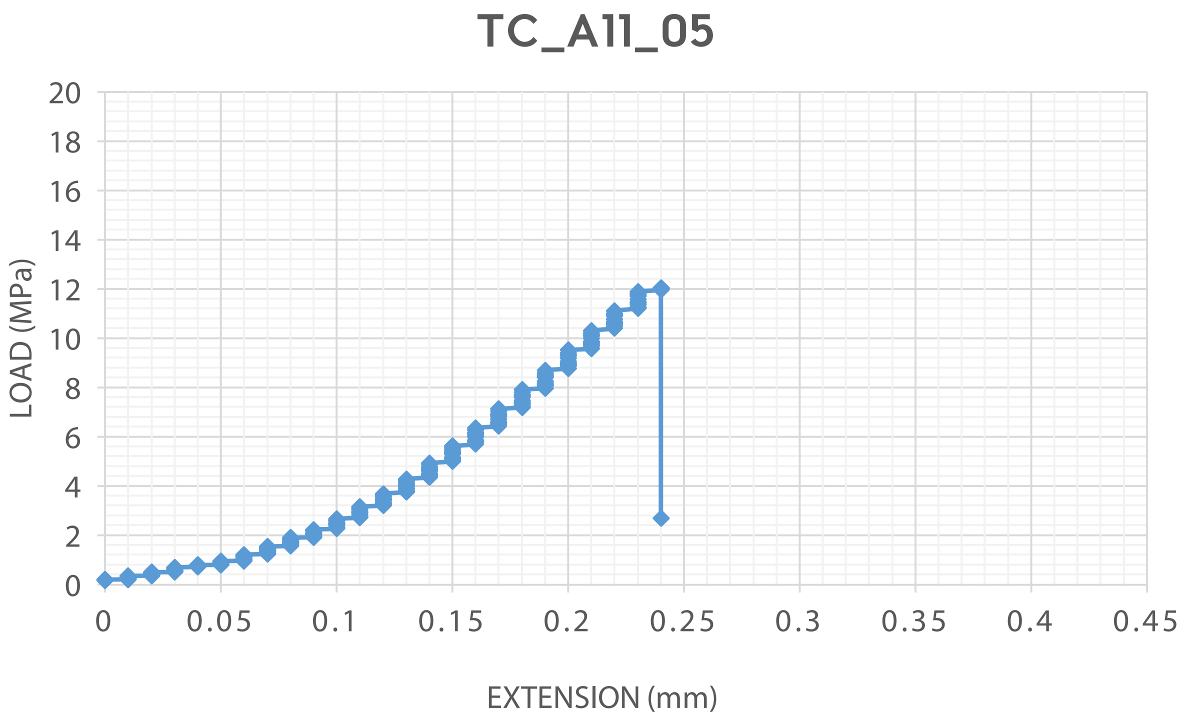
0.2
0.25
0.3
0.35
0.4
0.45


TC_A11_05
LOAD (MPa)
EXTENSION (mm)
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0.2
0.25
0.3
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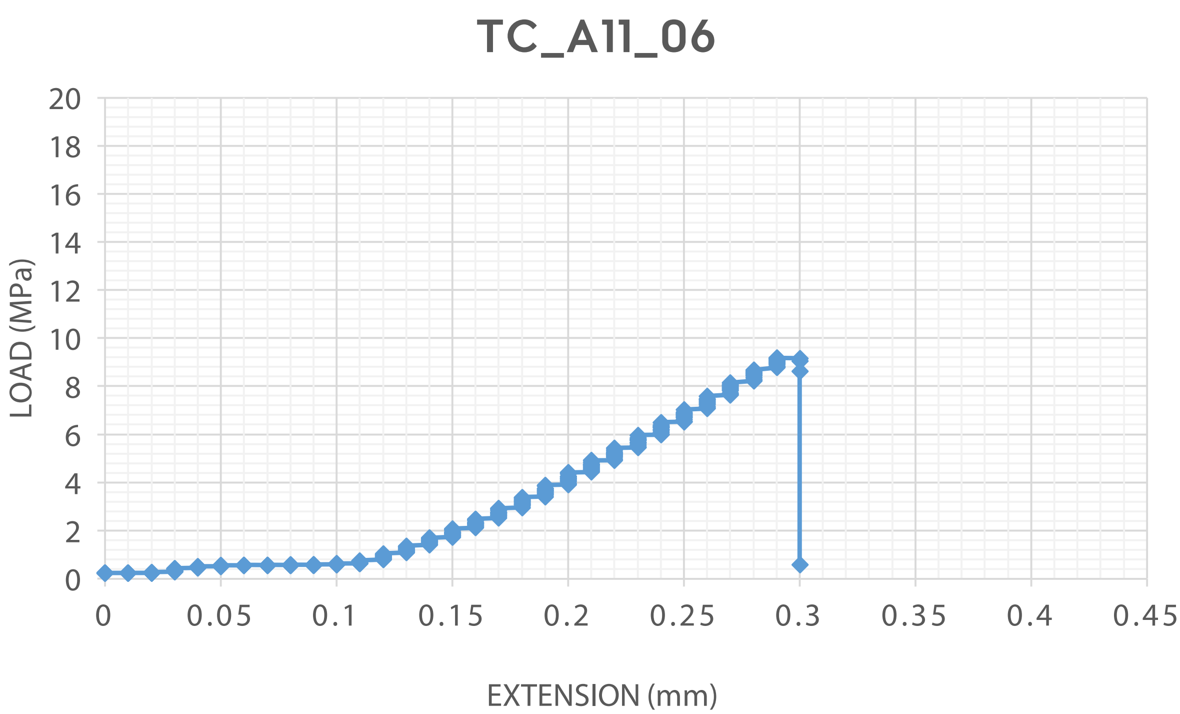
0.4
0.45


TC_A11_06
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EXTENSION (mm)
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0.15
0.2
0.25
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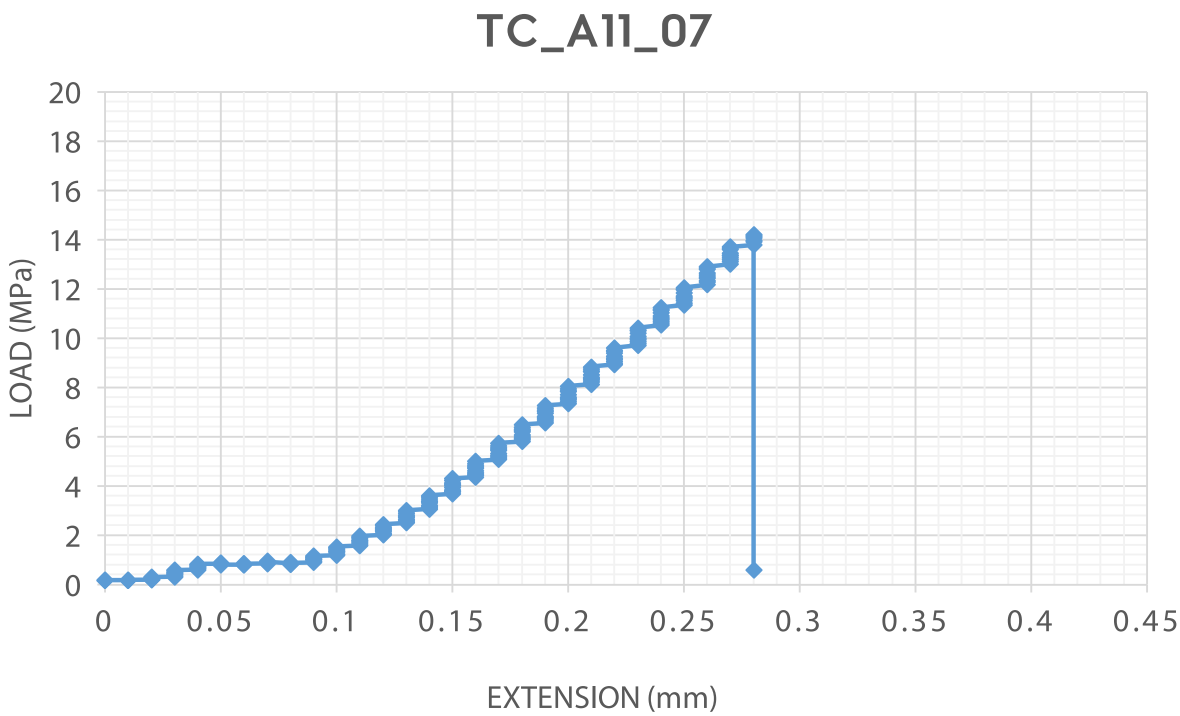
TC_A11_07
LOAD (MPa)
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0.1
0.15
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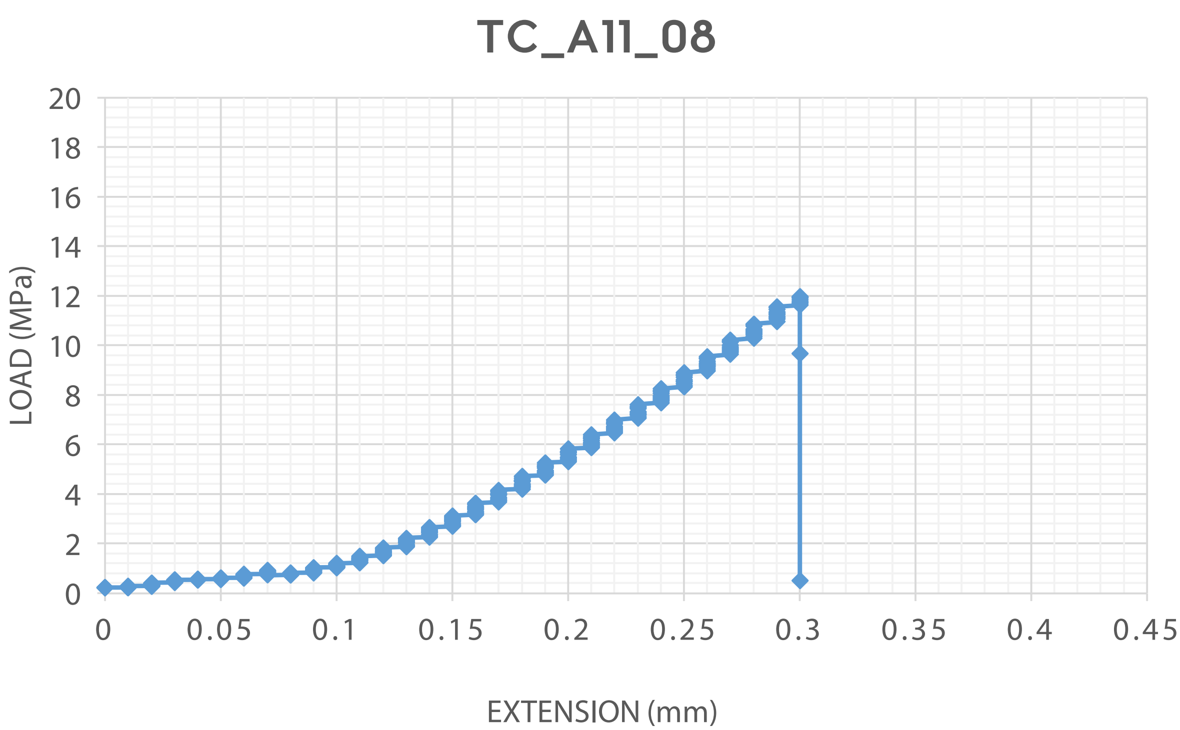
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_A11_08
LOAD (MPa)
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0.15
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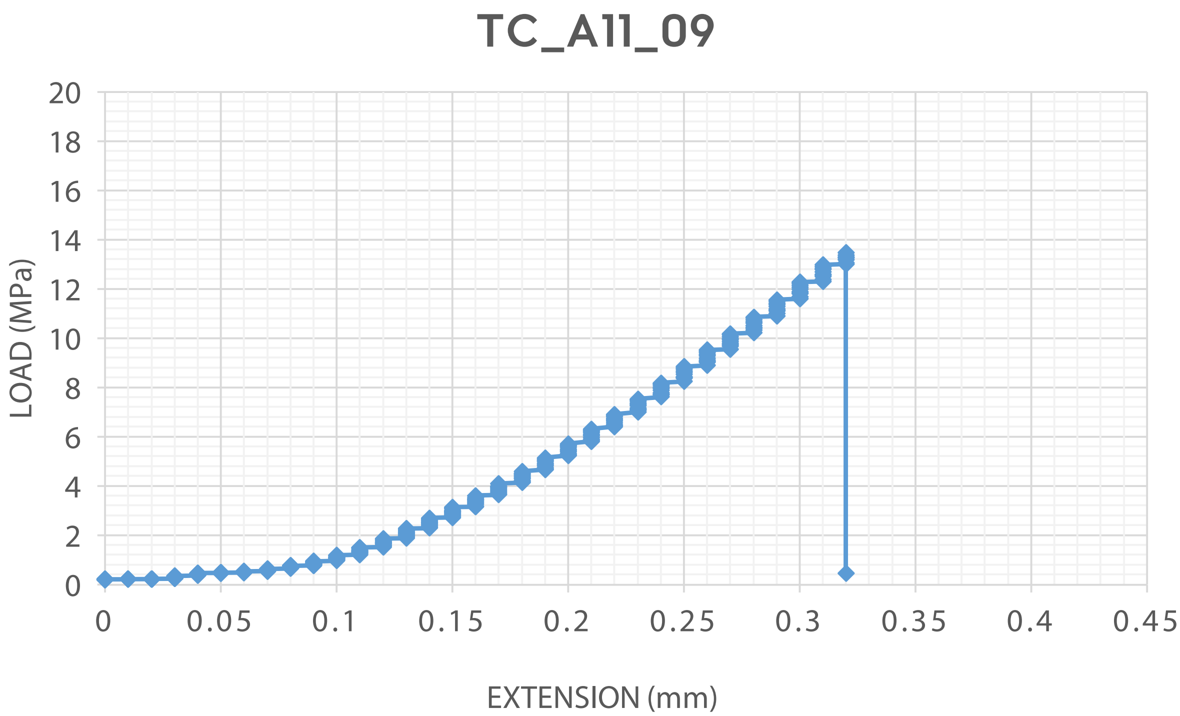
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_A11_09
LOAD (MPa)
EXTENSION (mm)
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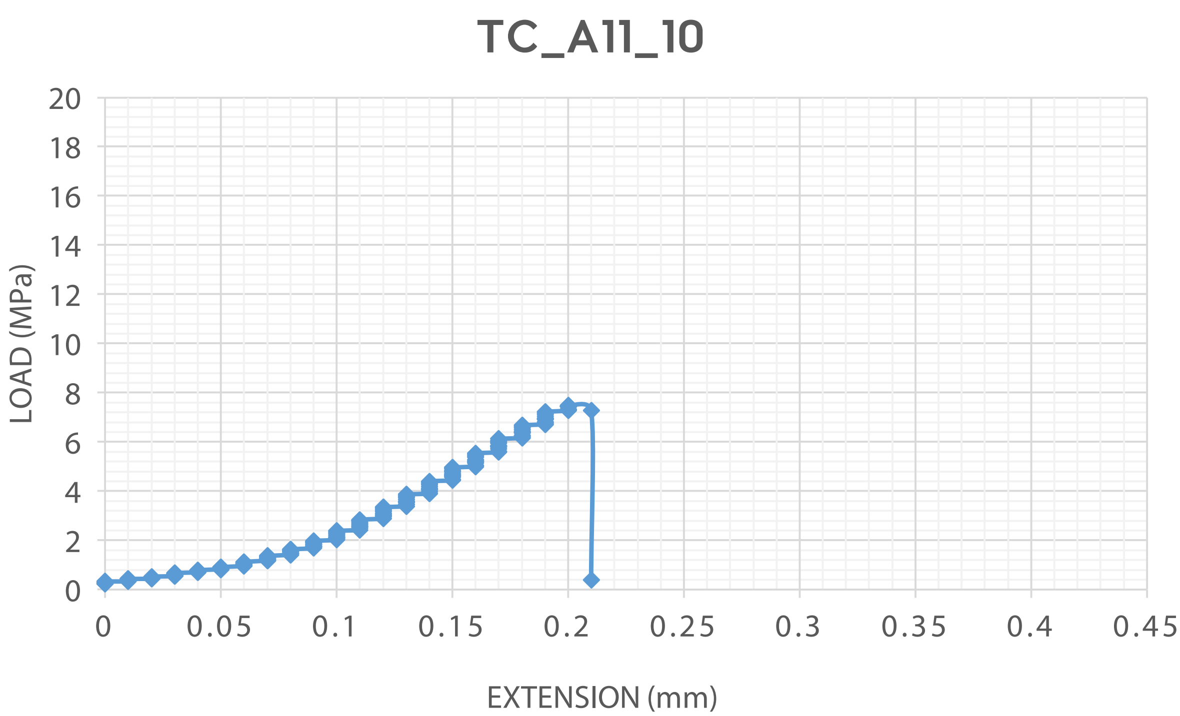
0.45


TC_A11_10
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EXTENSION (mm)
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0.25
0.3
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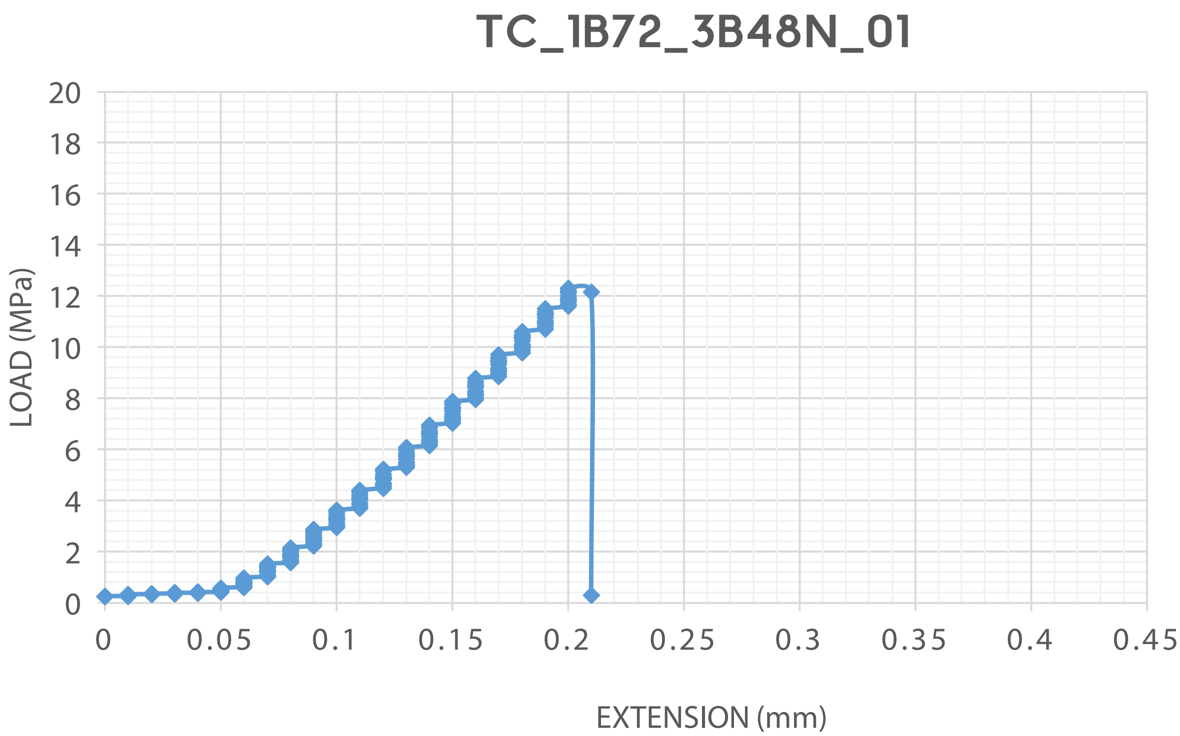
0.4
0.45


TC_1B72_3B48N_01
LOAD (MPa)
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0.35
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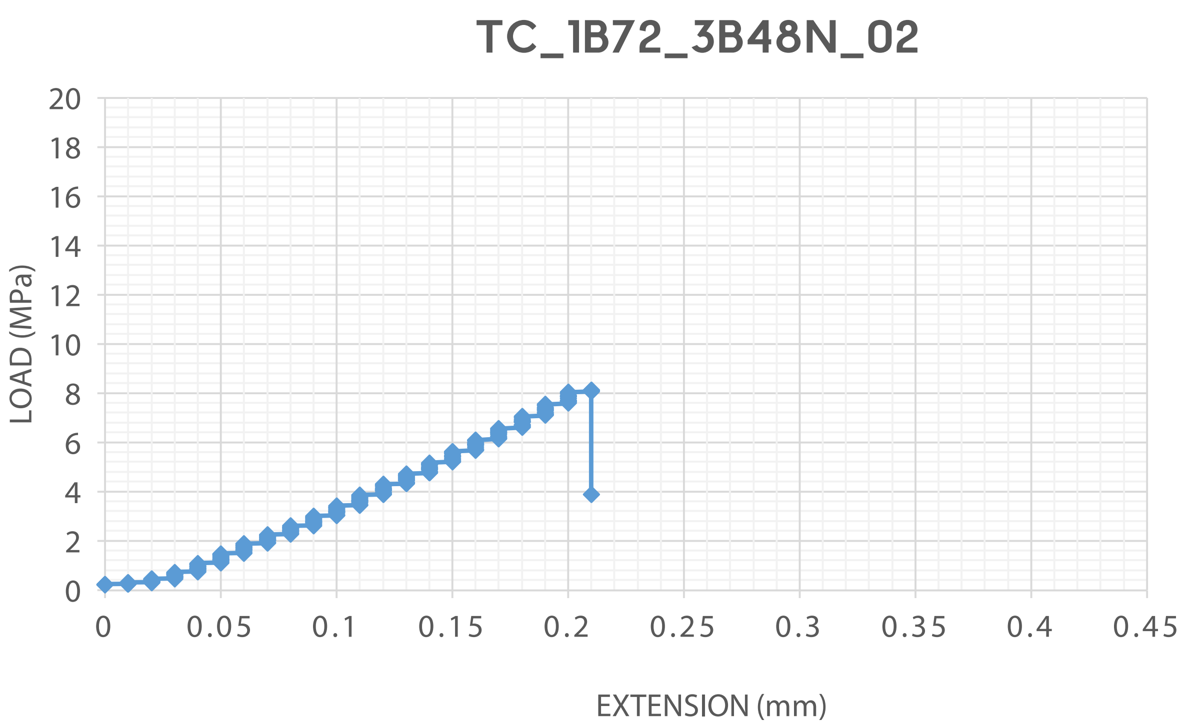
0.45
EXTENSION (mm)


TC_1B72_3B48N_02
LOAD (MPa)
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0.35
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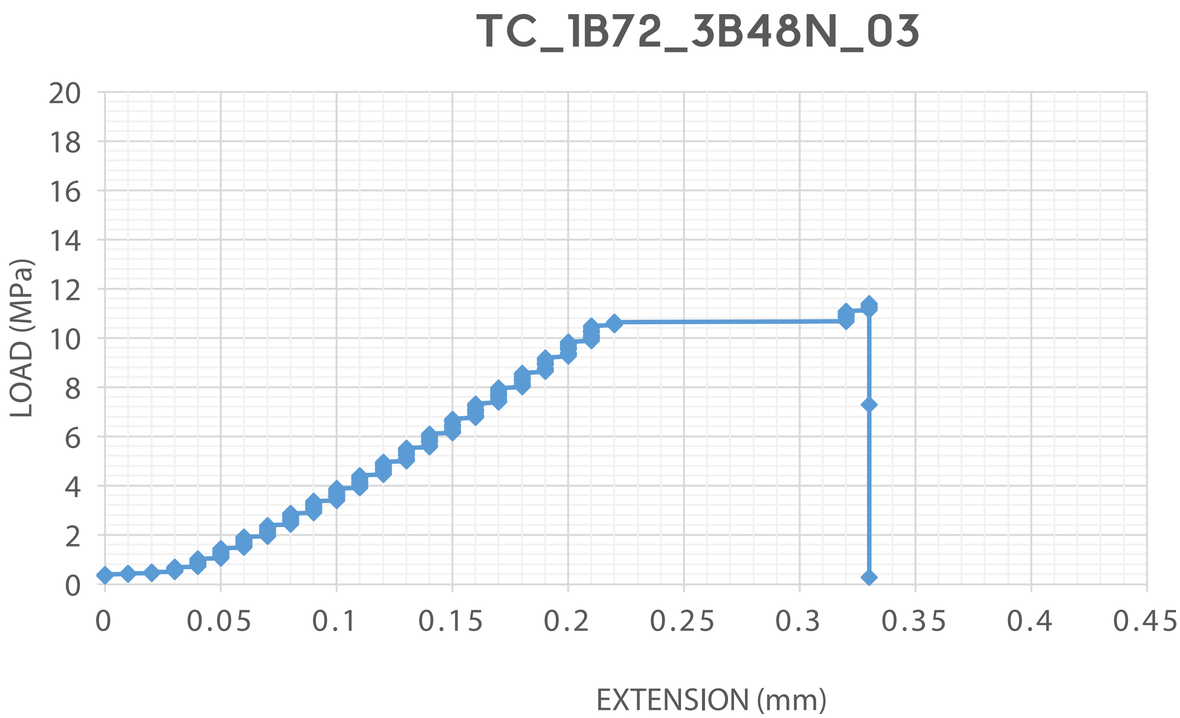
0.45
EXTENSION (mm)



TC_1B72_3B48N_03
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0.35
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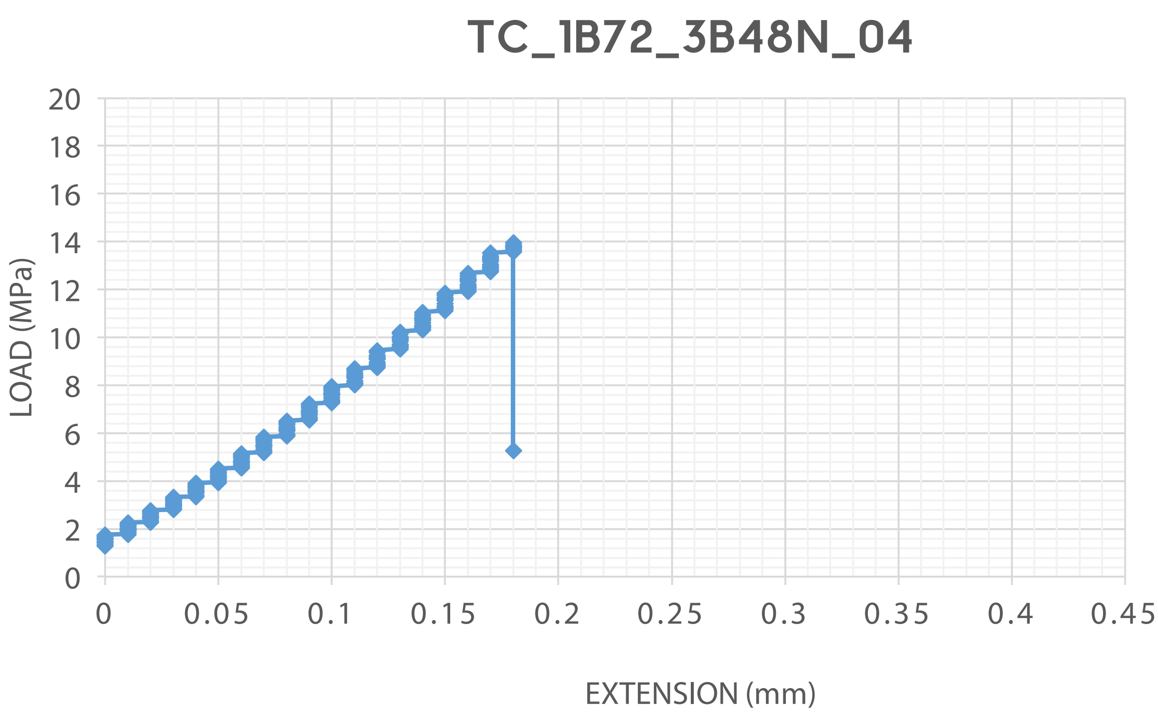
EXTENSION (mm)



TC_1B72_3B48N_04
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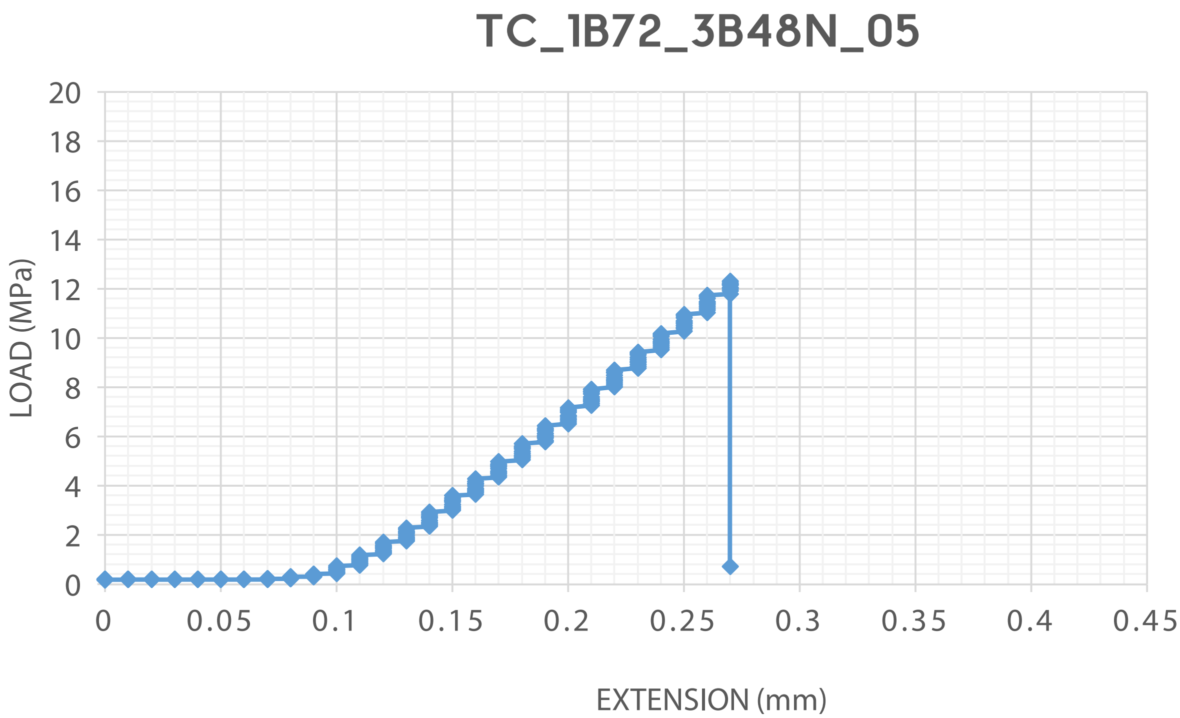
EXTENSION (mm)


TC_1B72_3B48N_05
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EXTENSION (mm)
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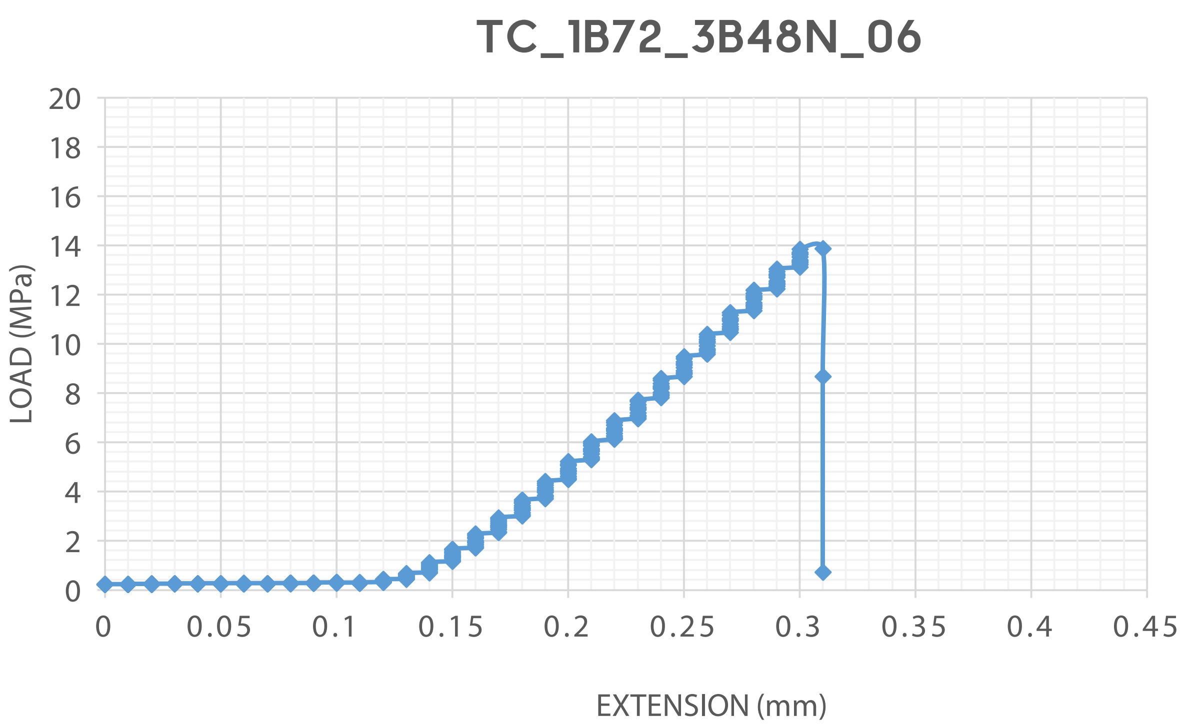
TC_1B72_3B48N_06
LOAD (MPa)
EXTENSION (mm)
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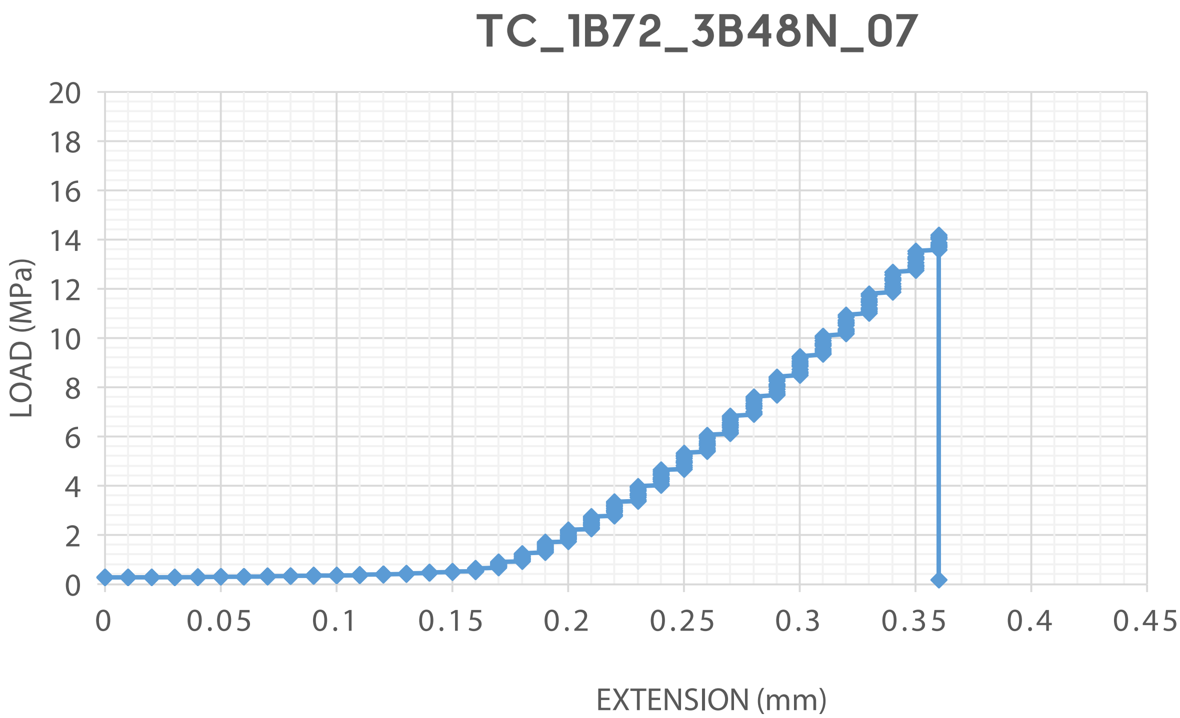
TC_1B72_3B48N_07
LOAD (MPa)
EXTENSION (mm)
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0.15
0.2
0.25
0.3
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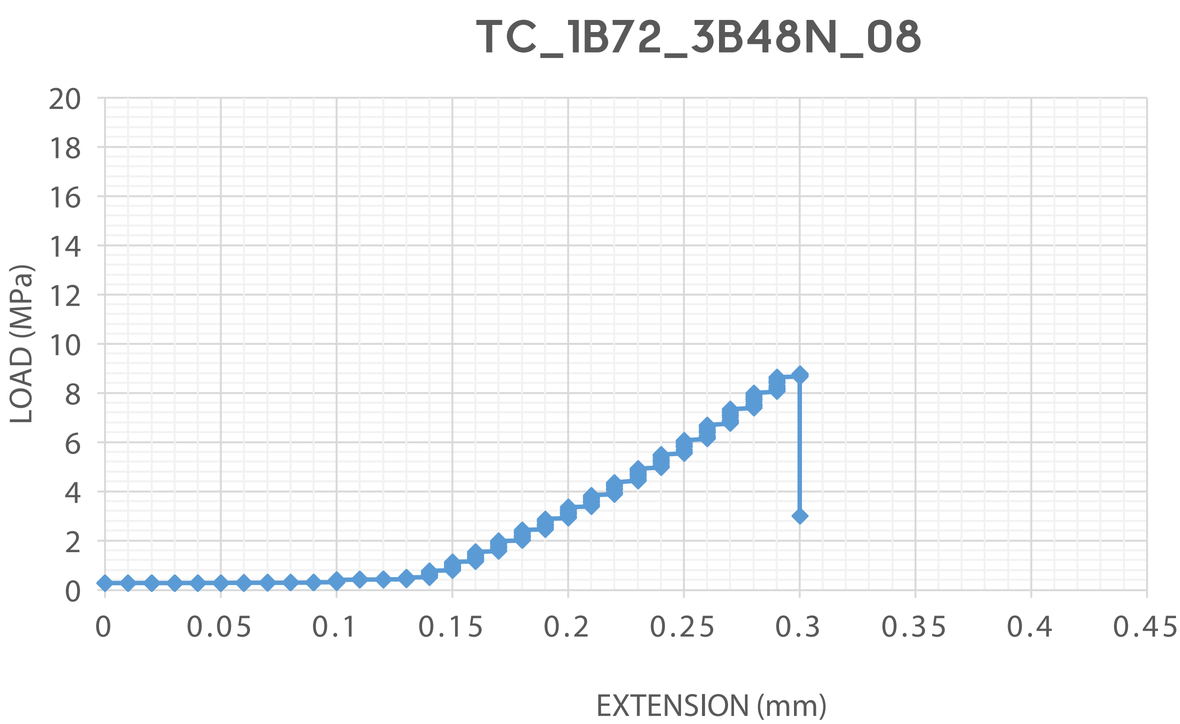
0.45


TC_1B72_3B48N_08
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0.3
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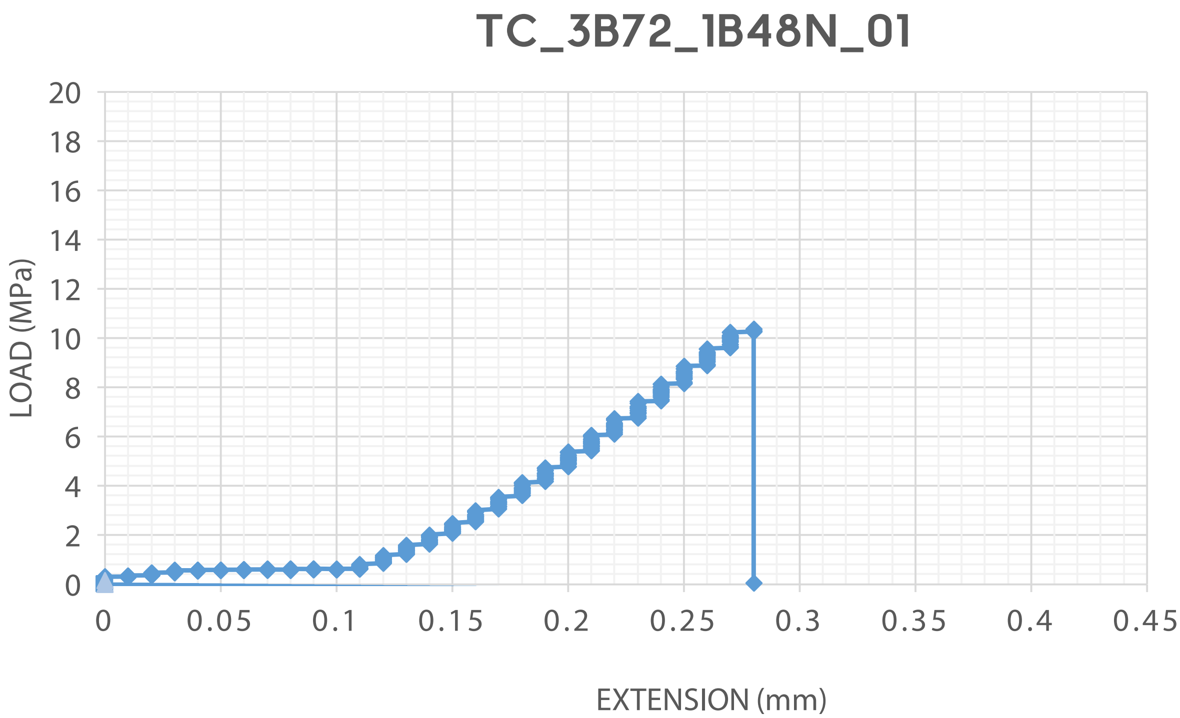
0.4
0.45


TC_3B72_1B48N_01
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0.15
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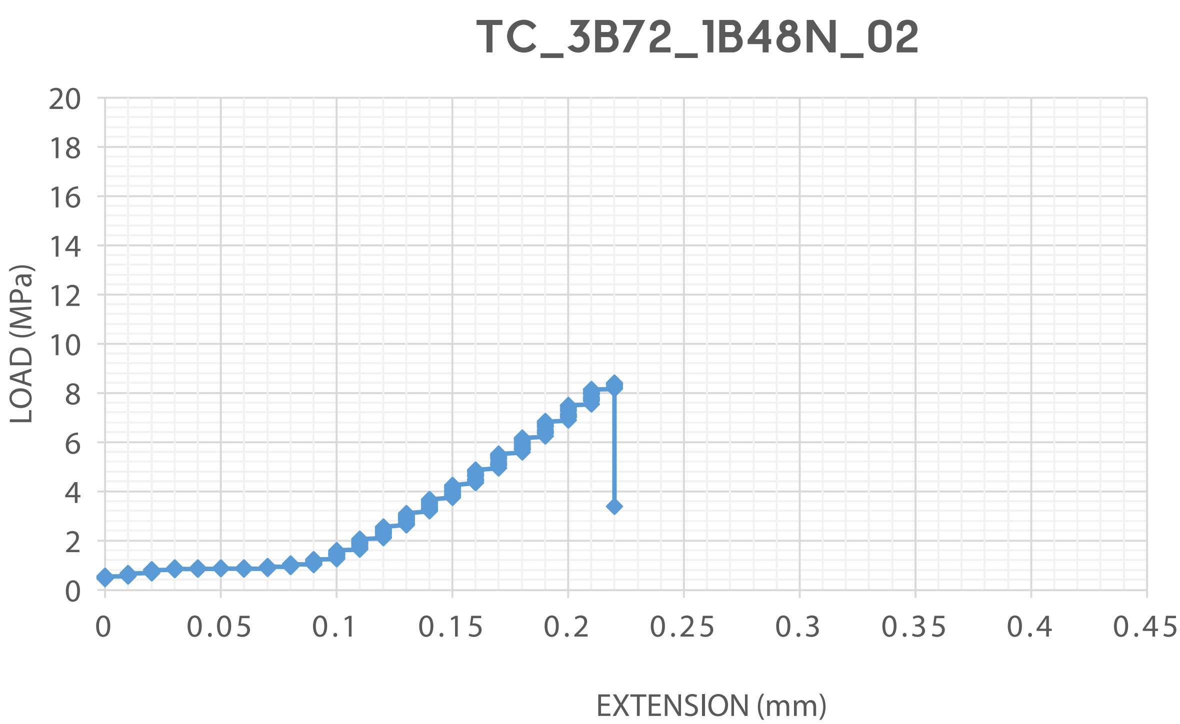
EXTENSION (mm)


TC_3B72_1B48N_02
LOAD (MPa)
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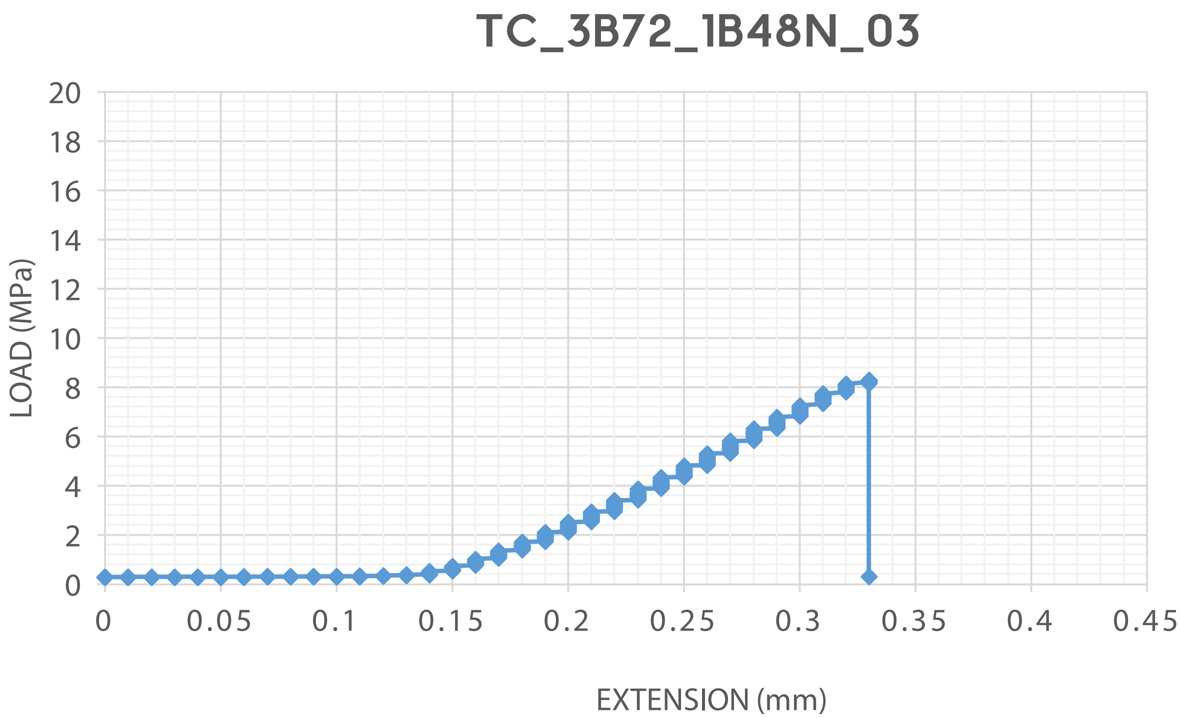
EXTENSION (mm)



TC_3B72_1B48N_03
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0.1
0.15
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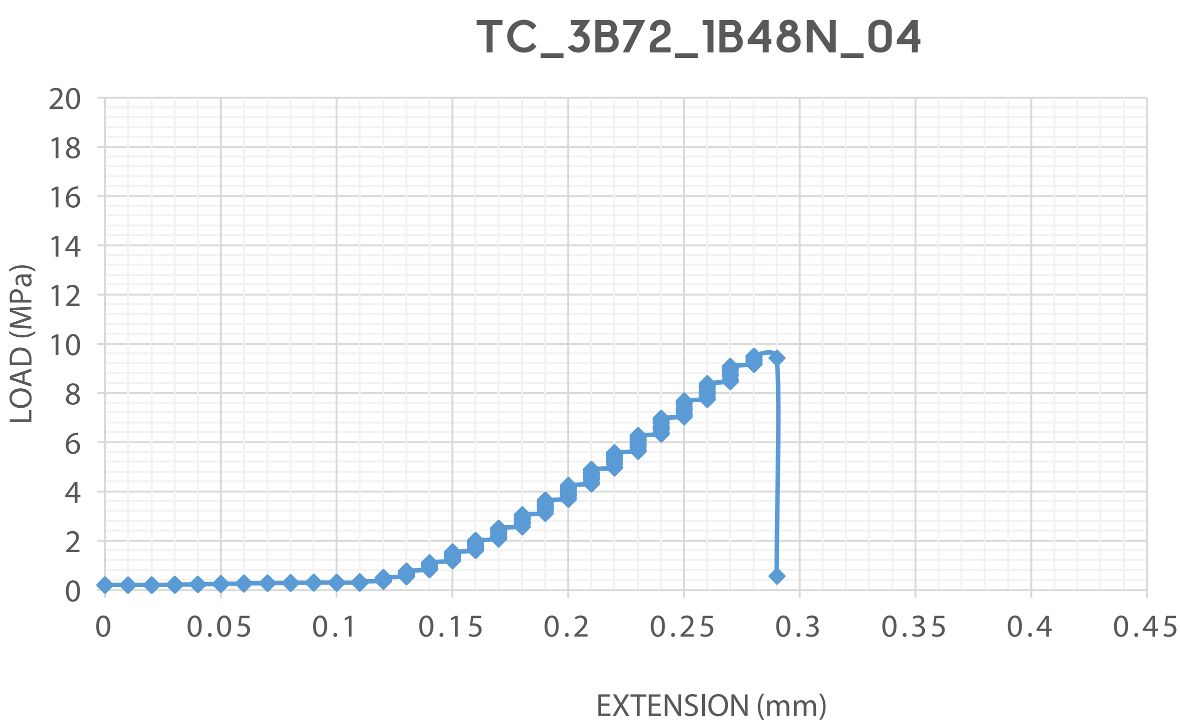
0.45
EXTENSION (mm)



TC_3B72_1B48N_04
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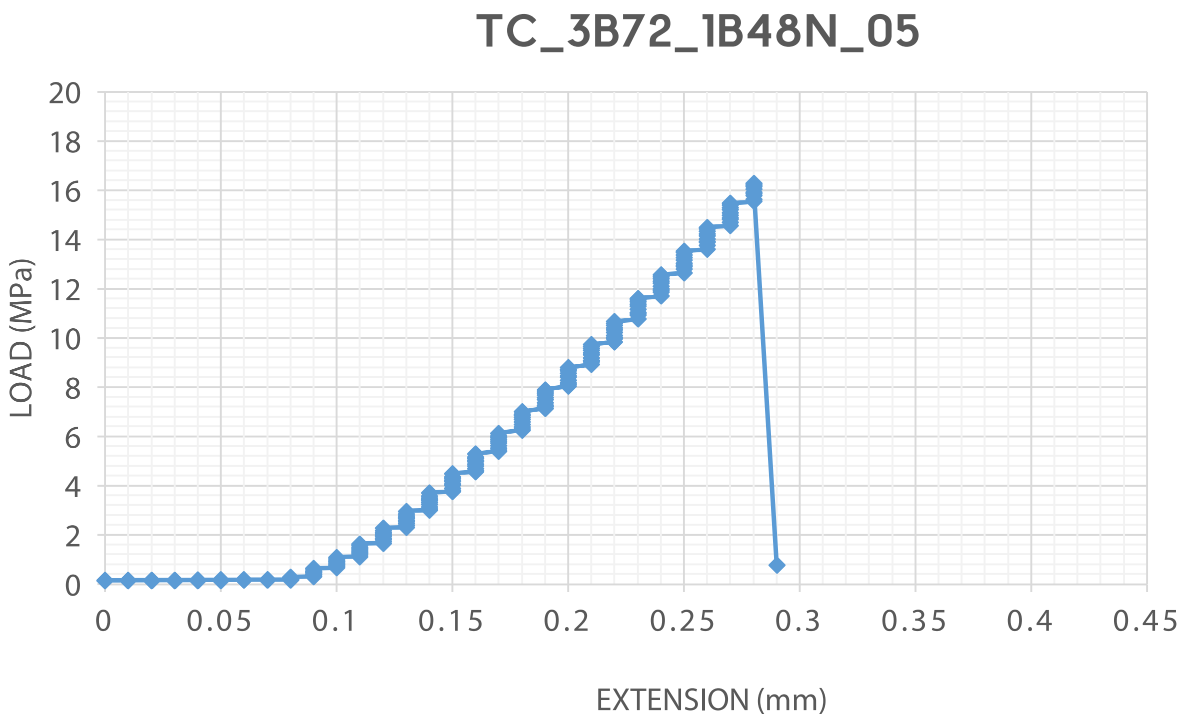
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_05
LOAD (MPa)
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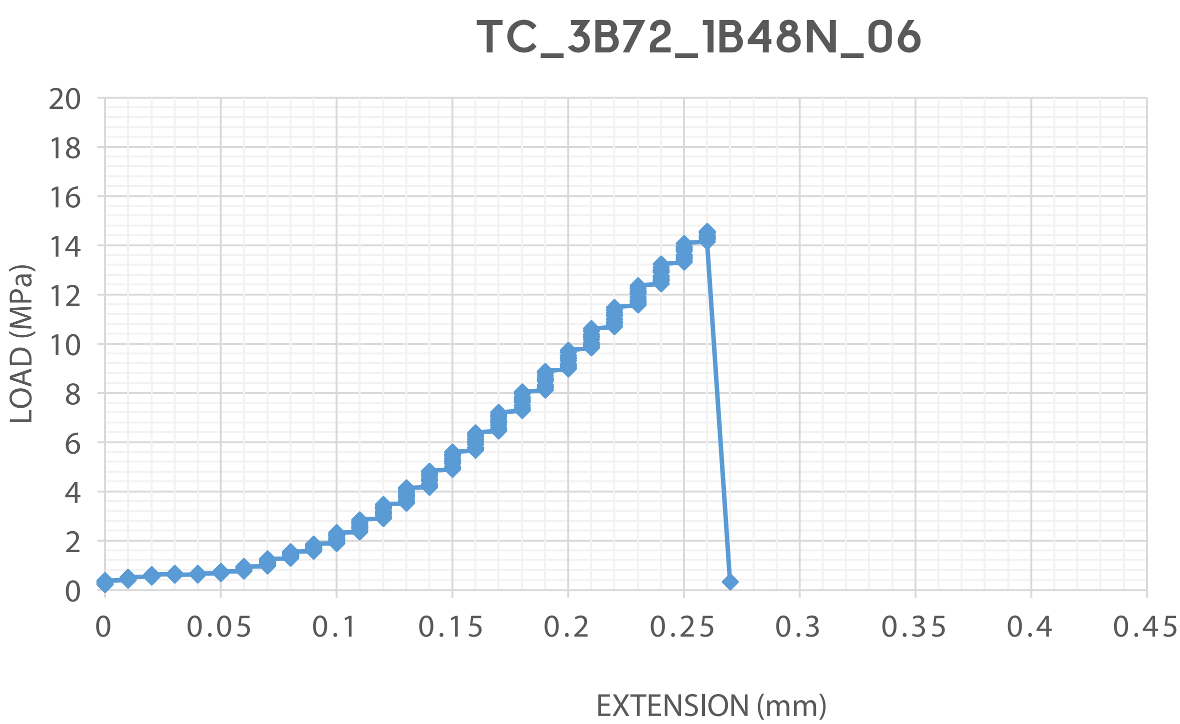
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_06
LOAD (MPa)
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6
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10
12
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16
18
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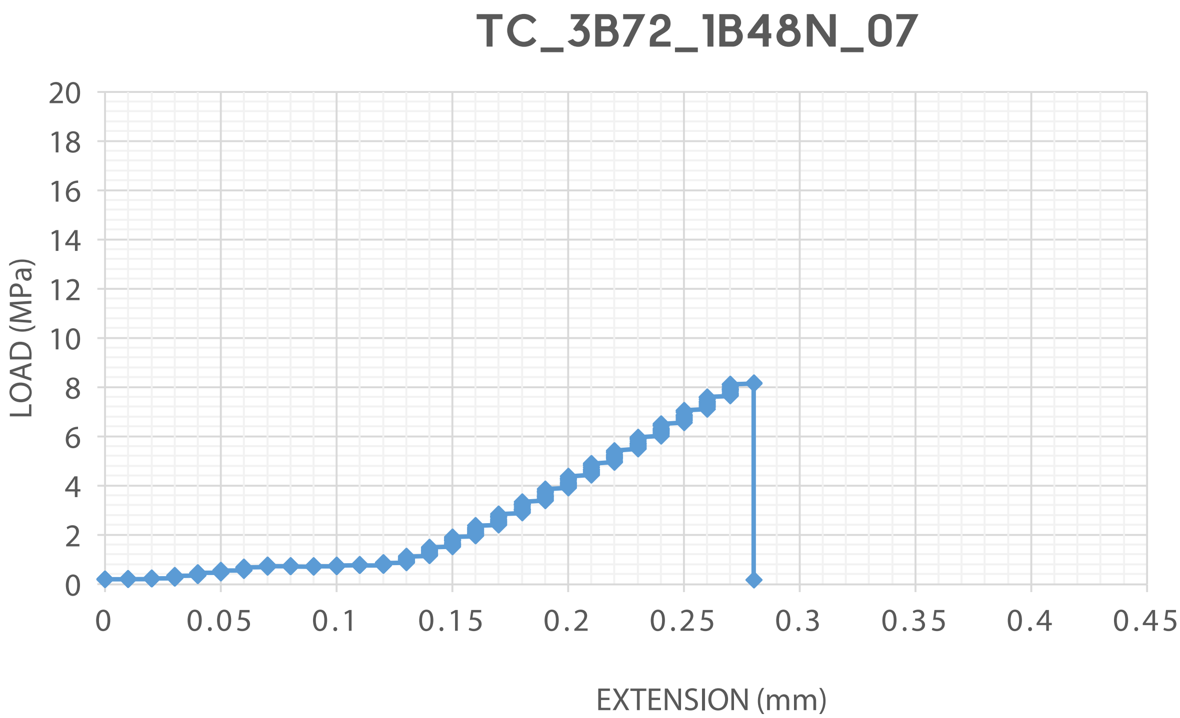
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)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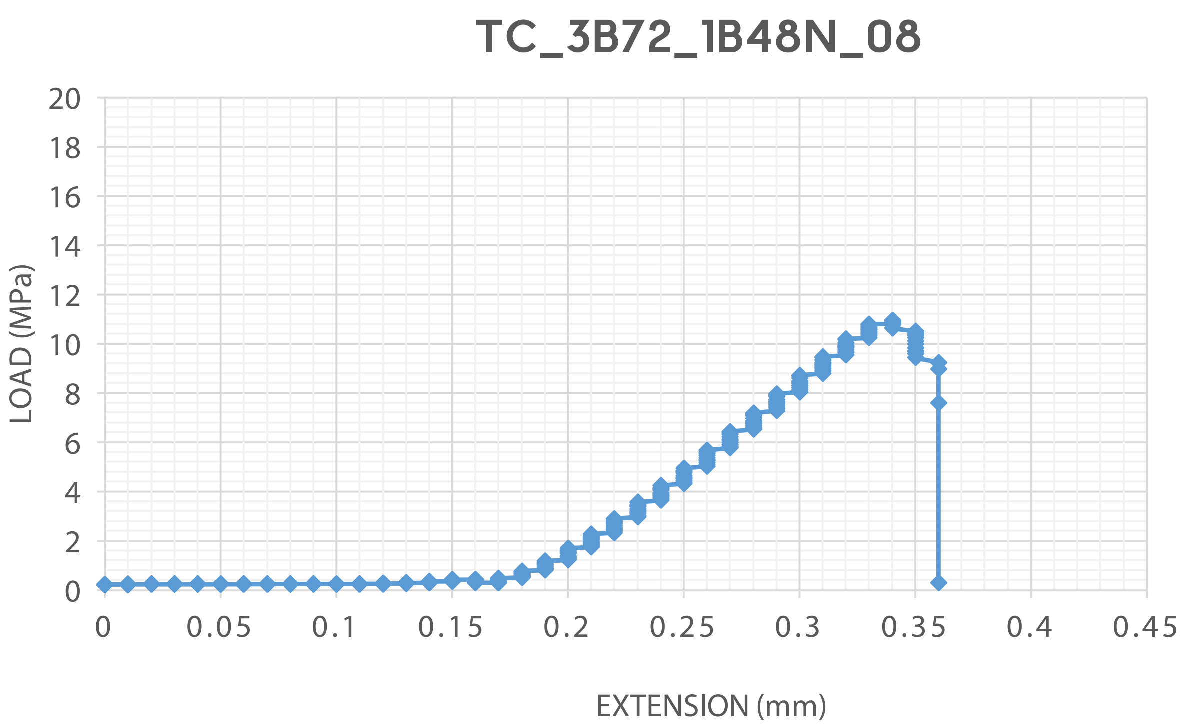
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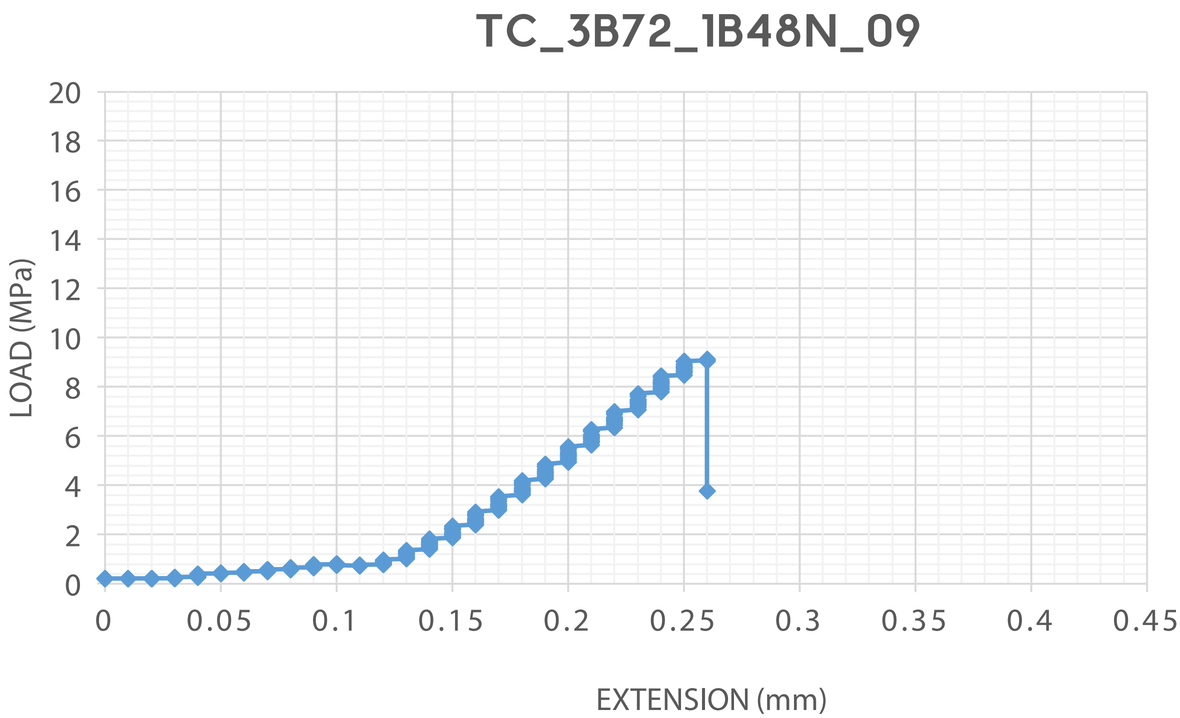
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0.25
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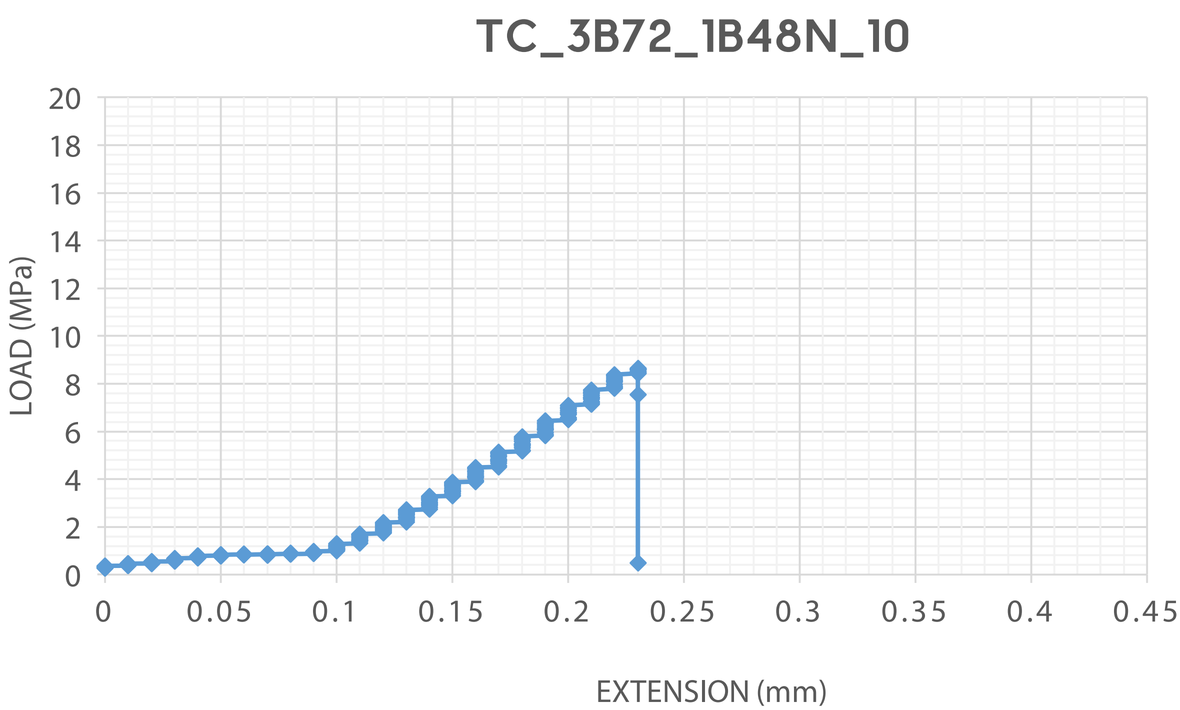
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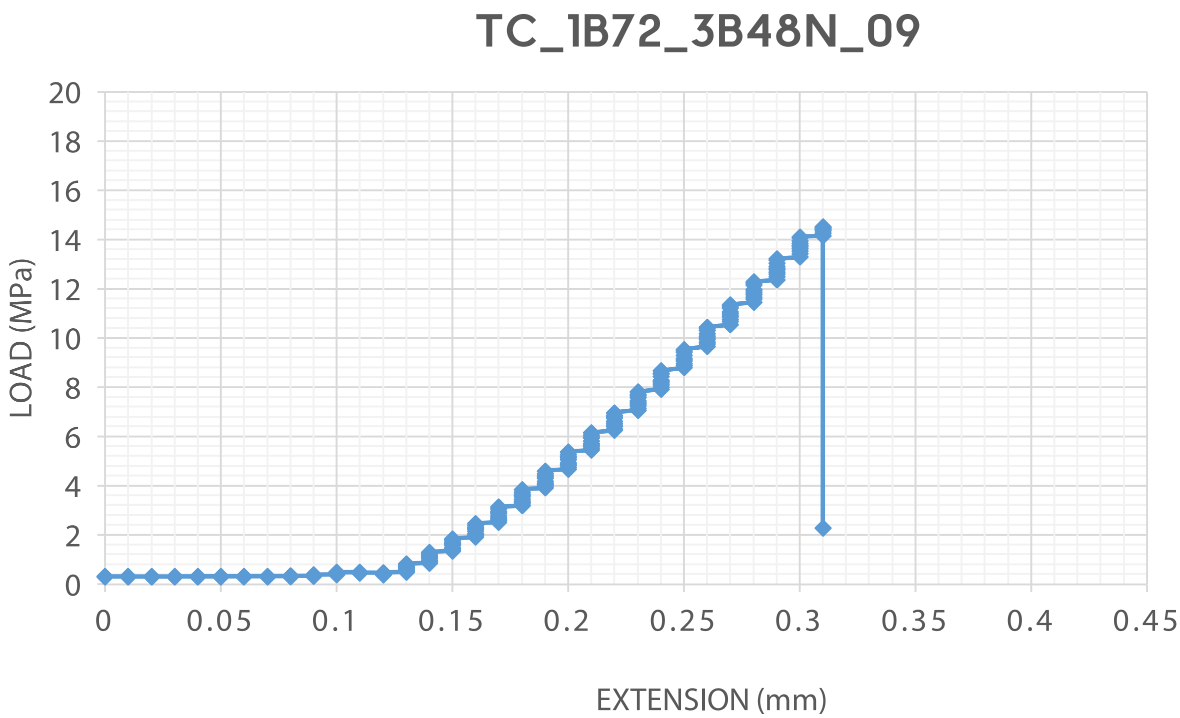
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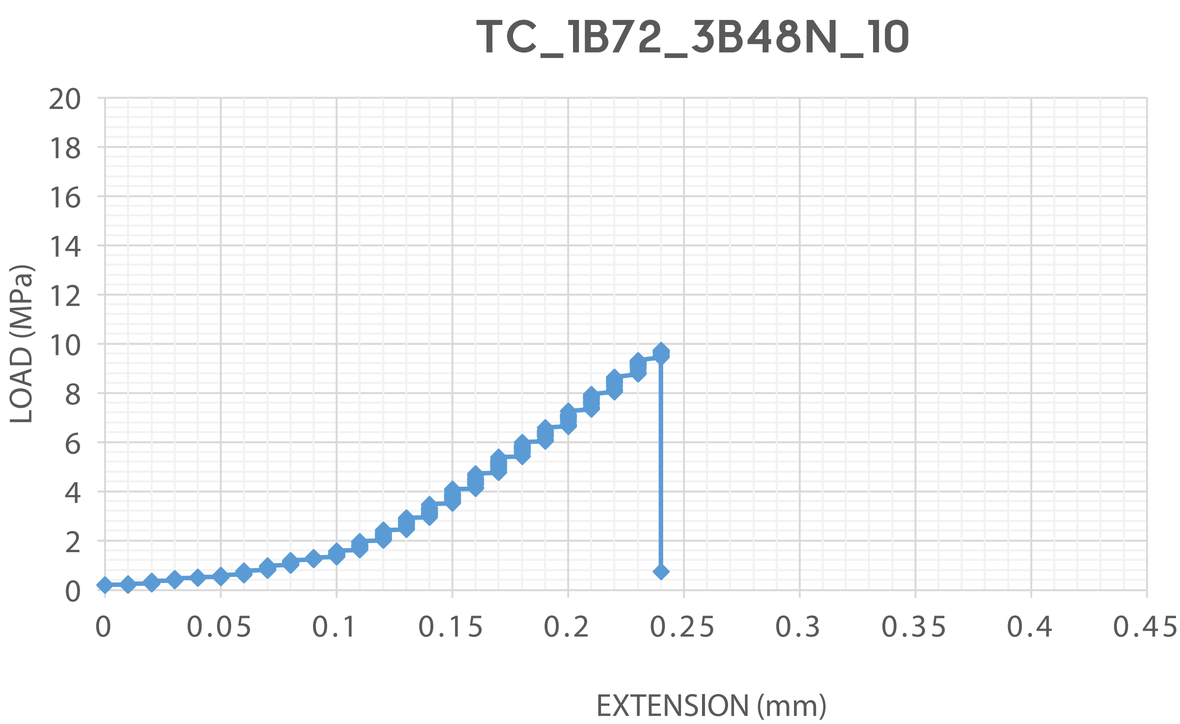
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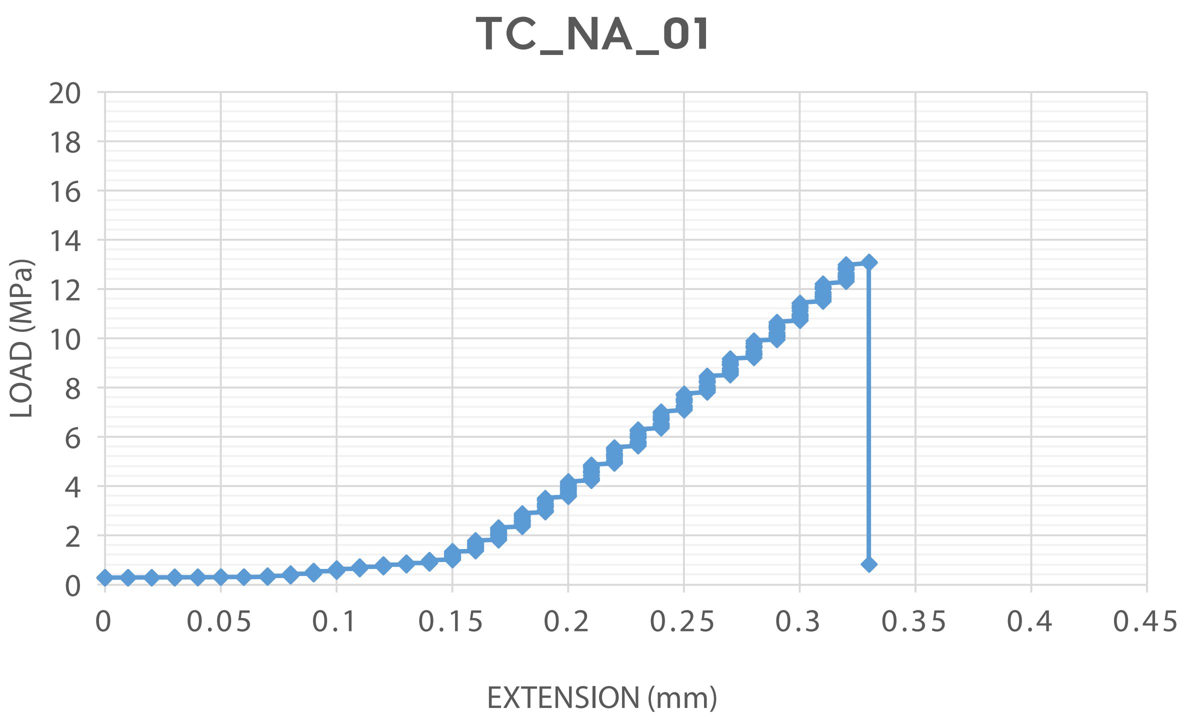
EXTENSION (mm)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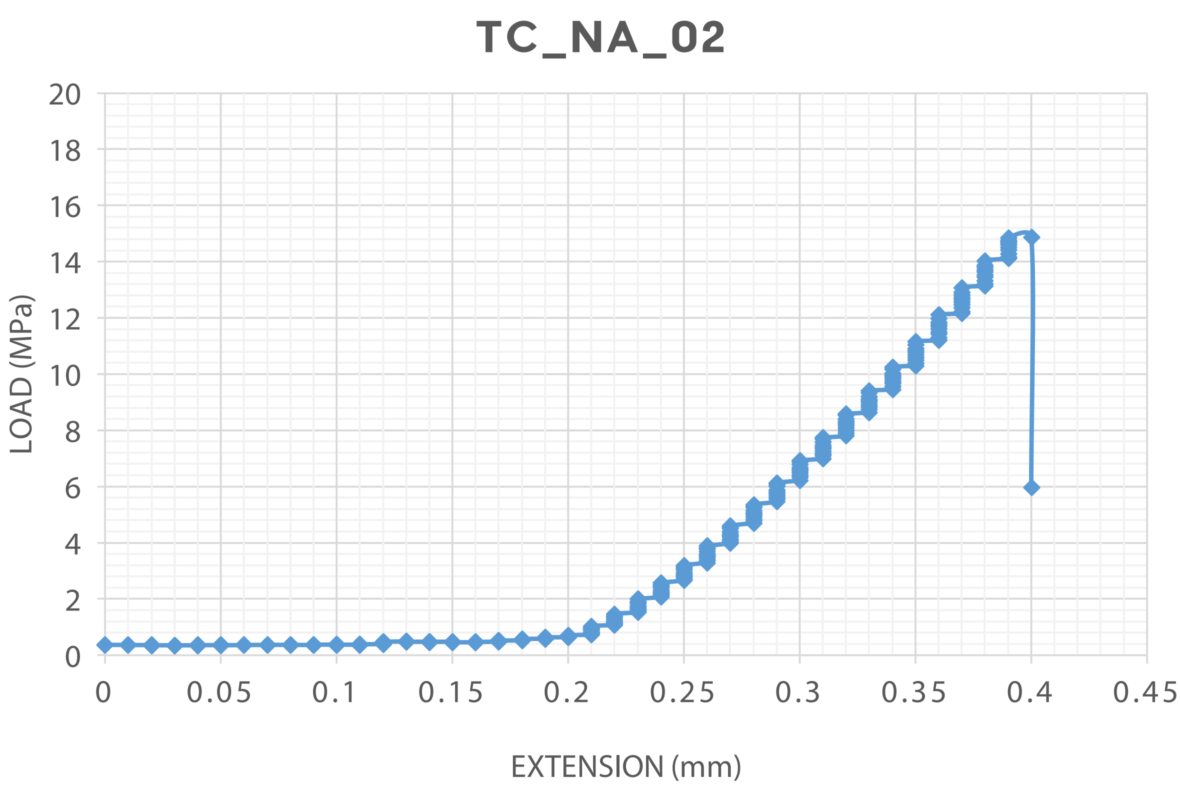
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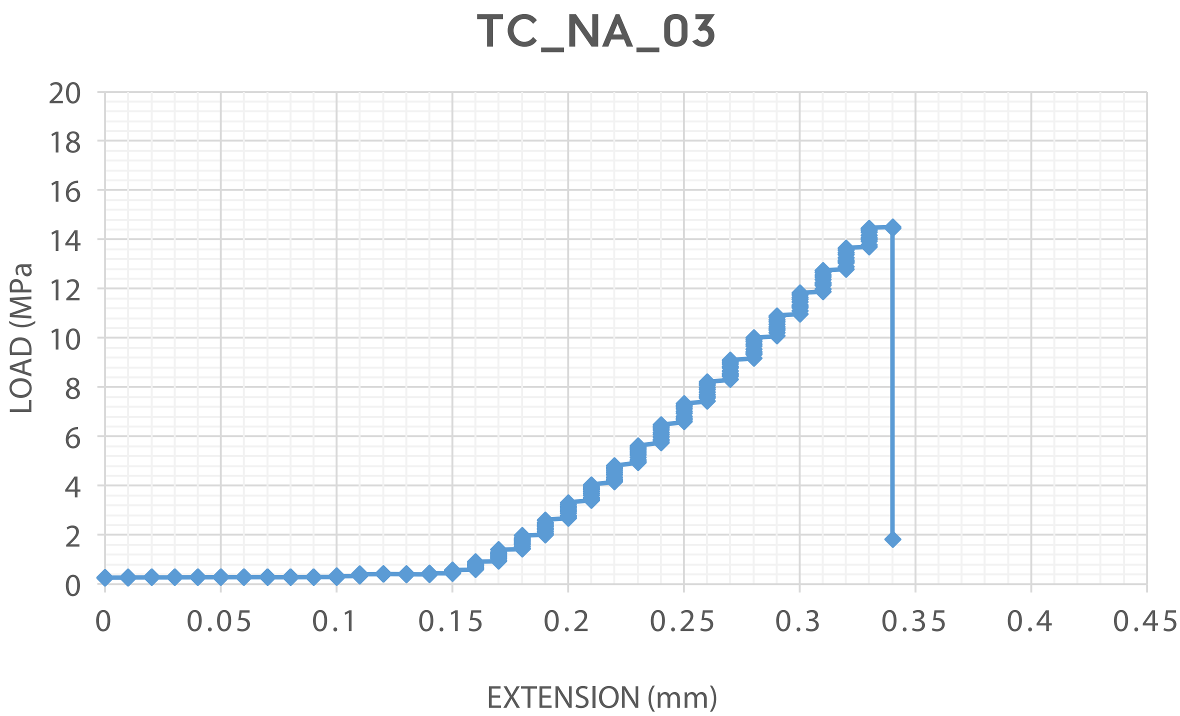
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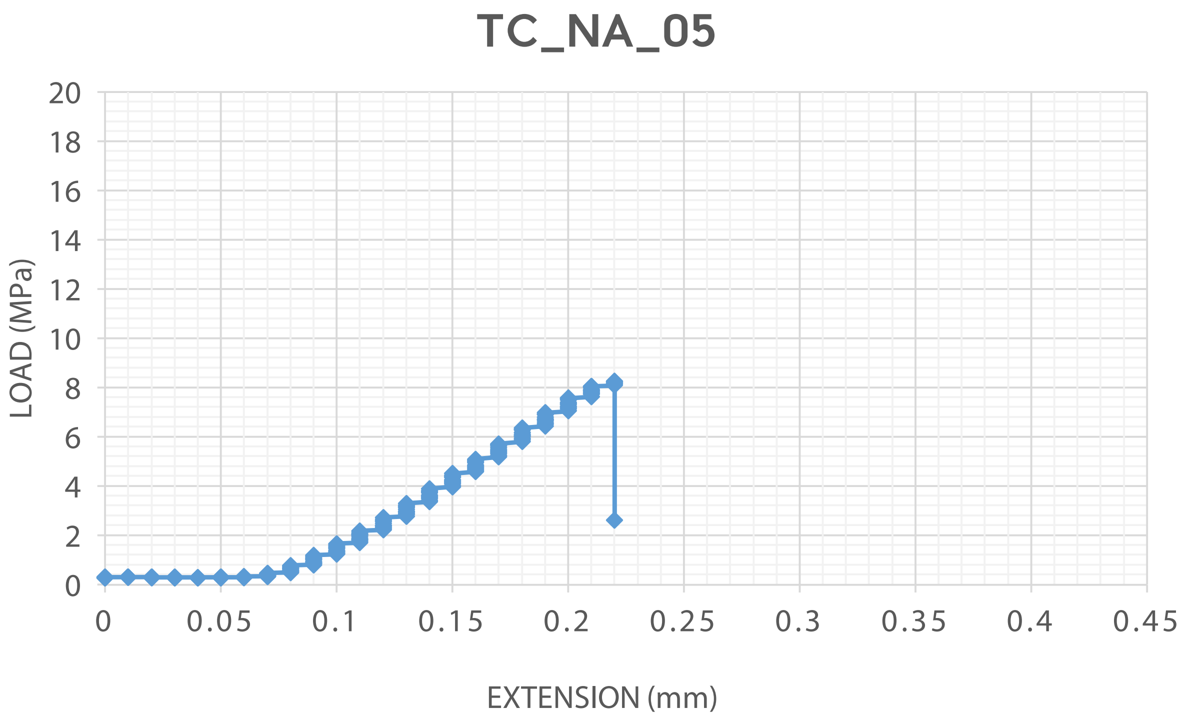
TC_NA_05
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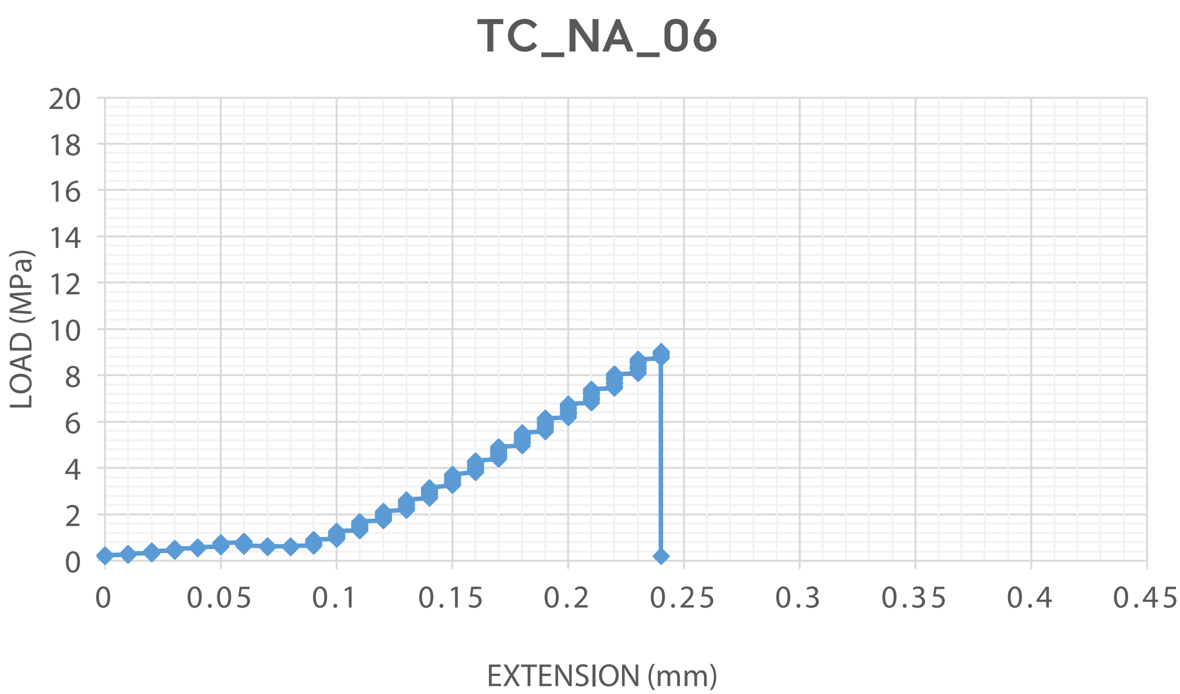
EXTENSION (mm)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0.1
0.15
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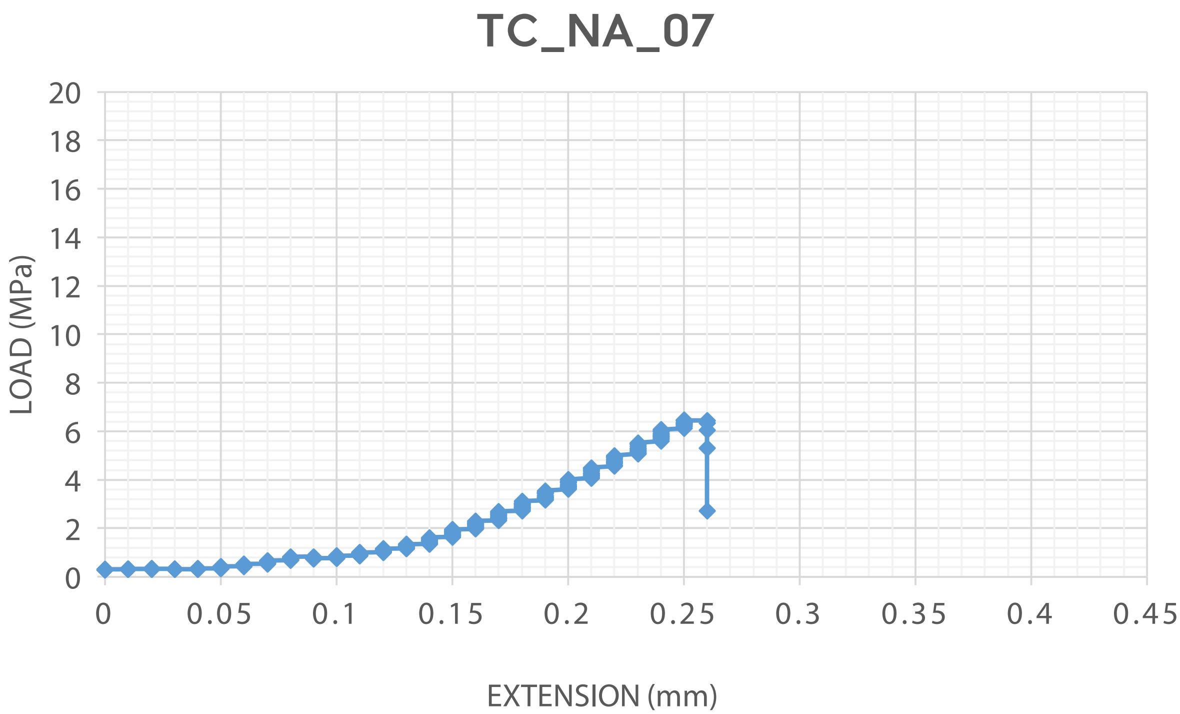
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)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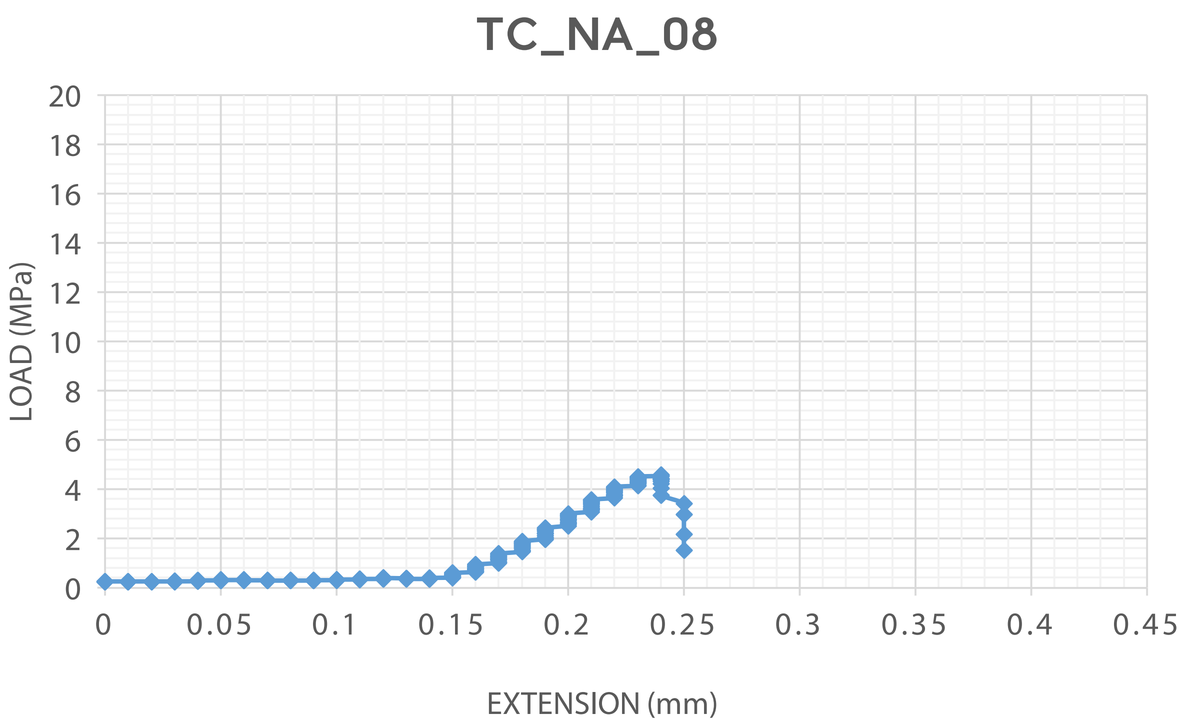
TC_NA_08
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EXTENSION (mm)
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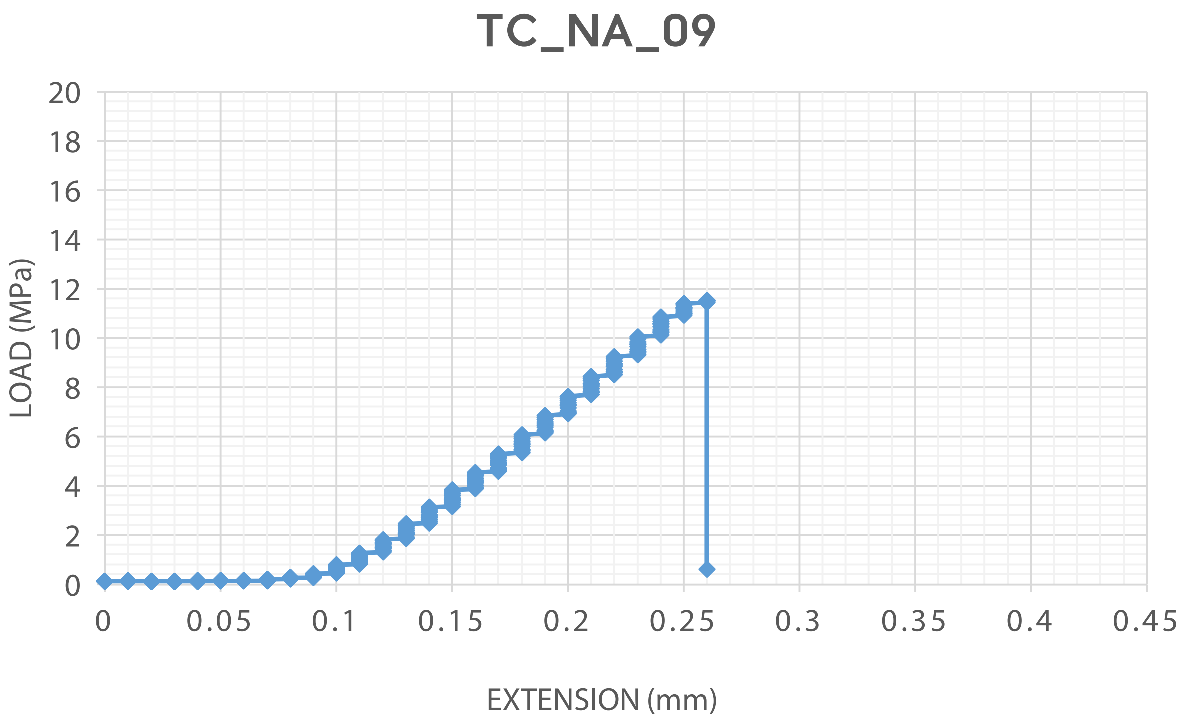
TC_NA_09
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0.1
0.15
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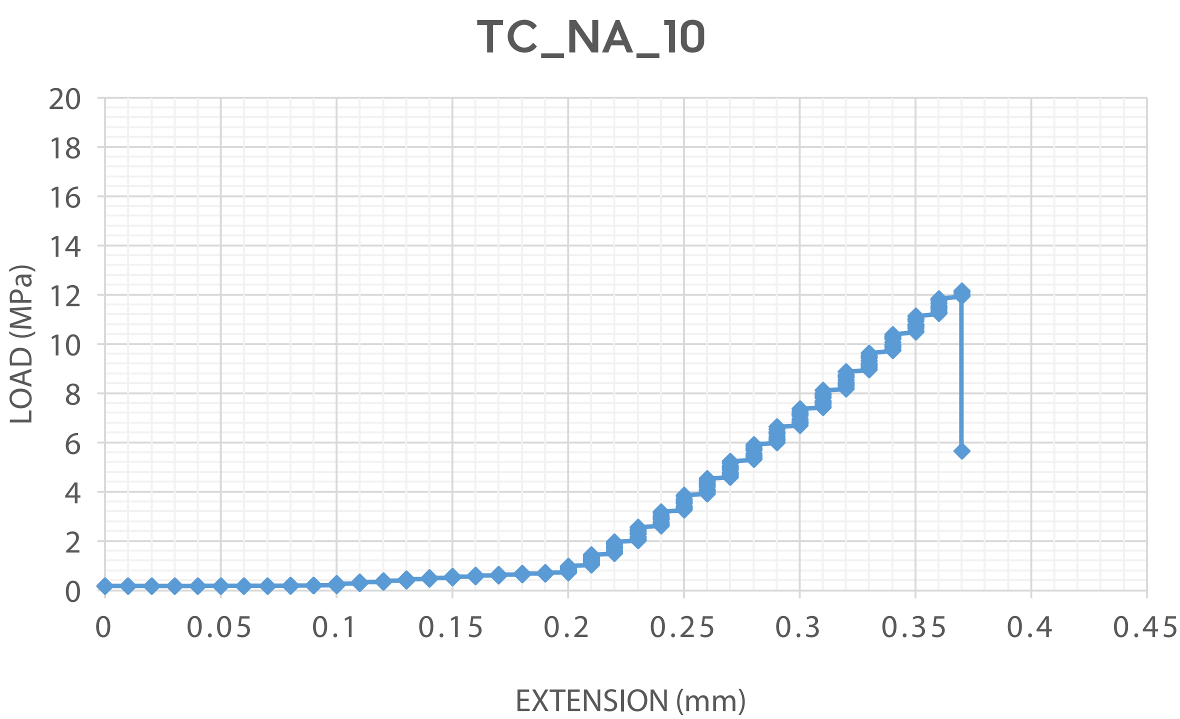
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NA_10
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0.05
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0.45
EXTENSION (mm)

APPENDIX 3

LIMESTONE – GROUP C

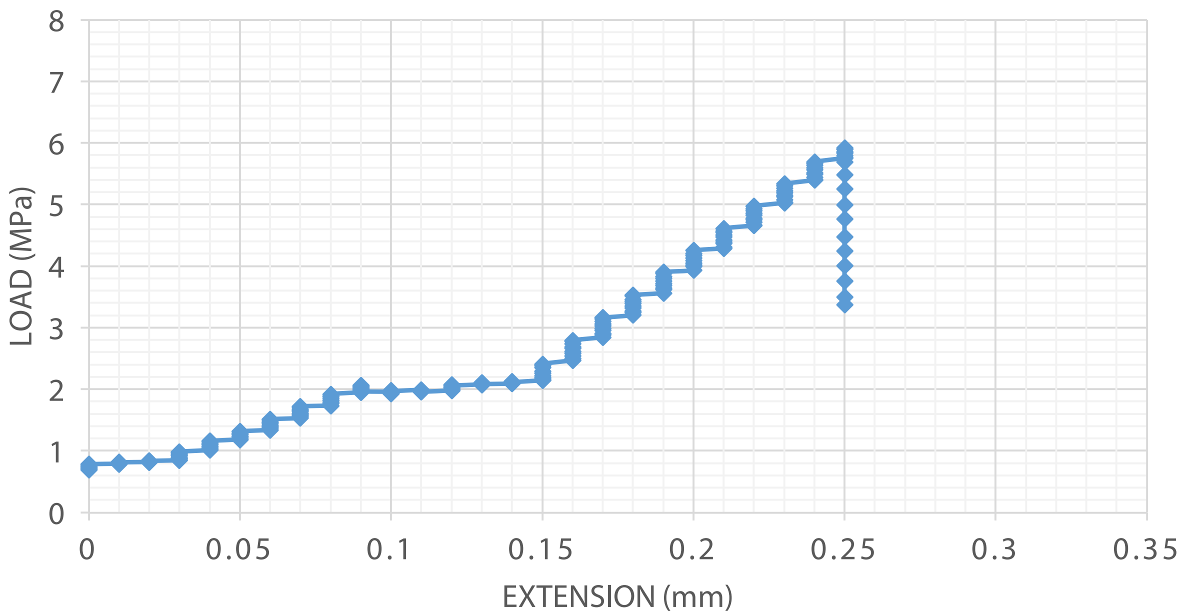
	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
L_B72_21	4/7/2017	4/24/2017	5/1/2017	369.67	Broke far from joint	6.06			1044.81
L_B72_22	4/7/2017	4/24/2017	5/1/2017	364.61	Broke next to joint	5.98			1027.50
L_B72_23	4/7/2017	4/24/2017	5/1/2017	376.63	Broke far from joint	6.18			988.24
L_B72_24	4/7/2017	4/24/2017	5/1/2017	384.13	Broke next to joint	6.30			1168.85
L_B72_25	4/7/2017	4/24/2017	5/1/2017	389.32	Broke next to joint	6.38			1509.14
L_B72_26	4/7/2017	4/24/2017	5/1/2017	384.13	Broke next to joint	6.30			1048.60
L_B72_27	4/7/2017	4/24/2017	5/1/2017	446.05	Broke next to joint	7.32			961.56
L_B72_28	4/7/2017	4/24/2017	5/1/2017	425.12	Broke next to joint	6.97			1185.47
L_B72_29	4/7/2017	4/24/2017	5/1/2017	444.03	Broke next to joint	7.28			1171.70
L_B72_30	4/7/2017	4/24/2017	5/1/2017	82.75	Broke next to joint	1.36			377.51
							6.01	1.71	
L_B48N_21	4/7/2017	4/24/2017	5/1/2017	440.91	Broke next to joint	7.23			981.35
L_B48N_22	4/7/2017	4/24/2017	5/1/2017	390.75	Broke far from joint	6.41			970.83
L_B48N_23	4/7/2017	4/24/2017	5/1/2017	351.05	Broke next to joint	5.76			744.39
L_B48N_24	4/7/2017	4/24/2017	5/1/2017	364.22	Broke next to joint	5.97			1024.60
L_B48N_25	4/7/2017	4/24/2017	5/1/2017	308.48	Broke next to joint	5.06			813.15
L_B48N_26	4/7/2017	4/24/2017	5/1/2017	445.99	No visible failure	7.31			900.15
L_B48N_27	4/7/2017	4/24/2017	5/1/2017	374.94	Broke next to joint	6.15			702.17
L_B48N_28	4/7/2017	4/24/2017	5/1/2017	434.8	Broke next to joint	7.13			792.99
L_B48N_29	4/7/2017	4/24/2017	5/1/2017	422.34	Broke next to joint	6.93			908.86
L_B48N_30	4/7/2017	4/24/2017	5/1/2017	451.63	Broke next to joint	7.41			821.42

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
							6.54	0.79	
L_B44_21	4/7/2017	4/24/2017	5/1/2017	396.13	No visible failure	6.50			1041.01
L_B44_22	4/7/2017	4/24/2017	5/1/2017	348.28	Broke next to joint	5.71			817.37
L_B44_23	4/7/2017	4/24/2017	5/1/2017	459.08	Broke next to joint	7.53			1164.90
L_B44_24	4/7/2017	4/24/2017	5/1/2017	374.28	No visible failure	6.14			1046.77
L_B44_25	4/7/2017	4/24/2017	5/1/2017	327.57	No visible failure	5.37			867.14
L_B44_26	4/7/2017	4/24/2017	5/1/2017	426.51	Broke next to joint	6.99			868.79
L_B44_27	4/7/2017	4/24/2017	5/1/2017	408.24	Broke next to joint	6.70			855.23
L_B44_28	4/7/2017	4/24/2017	5/1/2017	386.79	Broke next to joint	6.34			992.89
L_B44_29	4/7/2017	4/24/2017	5/1/2017	359.5	Broke next to joint	5.90			786.67
L_B44_30	4/7/2017	4/24/2017	5/1/2017	445.99	Broke next to joint	7.31			1001.58
							6.45	0.70	
L_A11_21	4/7/2017	4/24/2017	5/1/2017	391.23	No visible failure	6.42			699.37
L_A11_22	4/7/2017	4/24/2017	5/1/2017	311.42	No visible failure	5.11			2488.15
L_A11_23	4/7/2017	4/24/2017	5/1/2017	N/A	N/A	N/A			N/A
L_A11_24	4/7/2017	4/24/2017	5/1/2017	N/A	N/A	N/A			N/A
L_A11_25	4/7/2017	4/24/2017	5/1/2017	290.19	No visible failure	4.76			718.20
L_A11_26	4/7/2017	4/24/2017	5/1/2017	393.15	No visible failure	6.45			623.16
L_A11_27	4/7/2017	4/24/2017	5/1/2017	359.37	Broke next to joint	5.89			846.76
L_A11_28	4/7/2017	4/24/2017	5/1/2017	243.46	Broke next to joint	3.99			699.84
L_A11_29	4/7/2017	4/24/2017	5/1/2017	307.14	No visible failure	5.04			1486.35
L_A11_30	4/7/2017	4/24/2017	5/1/2017	326.06	Broke next to joint	5.35			1006.51

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
							5.38	0.84	
L_1B72_3B48N_21	4/7/2017	4/24/2017	5/1/2017	398.23	No visible failure	6.53			1159.30
L_1B72_3B48N_22	4/7/2017	4/24/2017	5/1/2017	453.96	Broke next to joint	7.44			1141.79
L_1B72_3B48N_23	4/7/2017	4/24/2017	5/1/2017	334.11	Broke next to joint	5.48			842.32
L_1B72_3B48N_24	4/7/2017	4/24/2017	5/1/2017	388.75	No visible failure	6.38			936.76
L_1B72_3B48N_25	4/7/2017	4/24/2017	5/1/2017	416.86	No visible failure	6.84			1174.54
L_1B72_3B48N_26	4/7/2017	4/24/2017	5/1/2017	449.89	Broke next to joint	7.38			1120.67
L_1B72_3B48N_27	4/7/2017	4/24/2017	5/1/2017	421.08	Broke next to joint	6.91			952.02
L_1B72_3B48N_28	4/7/2017	4/24/2017	5/1/2017	372.89	No visible failure	6.12			1019.28
L_1B72_3B48N_29	4/7/2017	4/24/2017	5/1/2017	378.6	No visible failure	6.21			919.69
L_1B72_3B48N_30	4/7/2017	4/24/2017	5/1/2017	428.80	No visible failure	7.03			946.46
							6.63	0.61	
L_3B72_1B48N_21	4/7/2017	4/24/2017	5/1/2017	391.83	Broke next to joint	6.43			1212.30
L_3B72_1B48N_22	4/7/2017	4/24/2017	5/1/2017	429.67	Broke next to joint	7.05			1262.25
L_3B72_1B48N_23	4/7/2017	4/24/2017	5/1/2017	412.87	Broke next to joint	6.77			1133.51
L_3B72_1B48N_24	4/7/2017	4/24/2017	5/1/2017	407.84	Broke next to joint	6.69			1285.34
L_3B72_1B48N_25	4/7/2017	4/24/2017	5/1/2017	366.96	Broke next to joint	6.02			1028.62
L_3B72_1B48N_26	4/7/2017	4/24/2017	5/1/2017	435.37	Broke next to joint	7.14			1412.88
L_3B72_1B48N_27	4/7/2017	4/24/2017	5/1/2017	418.01	Broke next to joint	6.86			1015.17
L_3B72_1B48N_28	4/7/2017	4/24/2017	5/1/2017	414.22	Broke next to joint	6.79			1110.51
L_3B72_1B48N_29	4/7/2017	4/24/2017	5/1/2017	426.75	Broke far from joint	7.00			935.99
L_3B72_1B48N_30	4/7/2017	4/24/2017	5/1/2017	409.64	Broke next to joint	6.72			1352.13

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
							6.75	0.33	
L_CT_NH_21	N/A	N/A	4/5/2017	478.66		7.85			1551.81
L_CT_NH_22	N/A	N/A	4/5/2017	423.84		6.95			1187.58
L_CT_NH_23	N/A	N/A	4/5/2017	426.39		6.99			1128.06
L_CT_NH_24	N/A	N/A	4/5/2017	455.81		7.48			1114.21
L_CT_NH_25	N/A	N/A	4/5/2017	429.63		7.05			1030.68
L_CT_NH_26	N/A	N/A	4/5/2017	437.38		7.17			1024.25
L_CT_NH_27	N/A	N/A	4/5/2017	477.78		7.84			1306.48
L_CT_NH_28	N/A	N/A	4/5/2017	427.96		7.02			1104.62
L_CT_NH_29	N/A	N/A	4/5/2017	471.21		7.73			1286.99
L_CT_NH_30	N/A	N/A	4/5/2017	447.97		7.35			1143.02
							7.34	0.36	

L_B72_21



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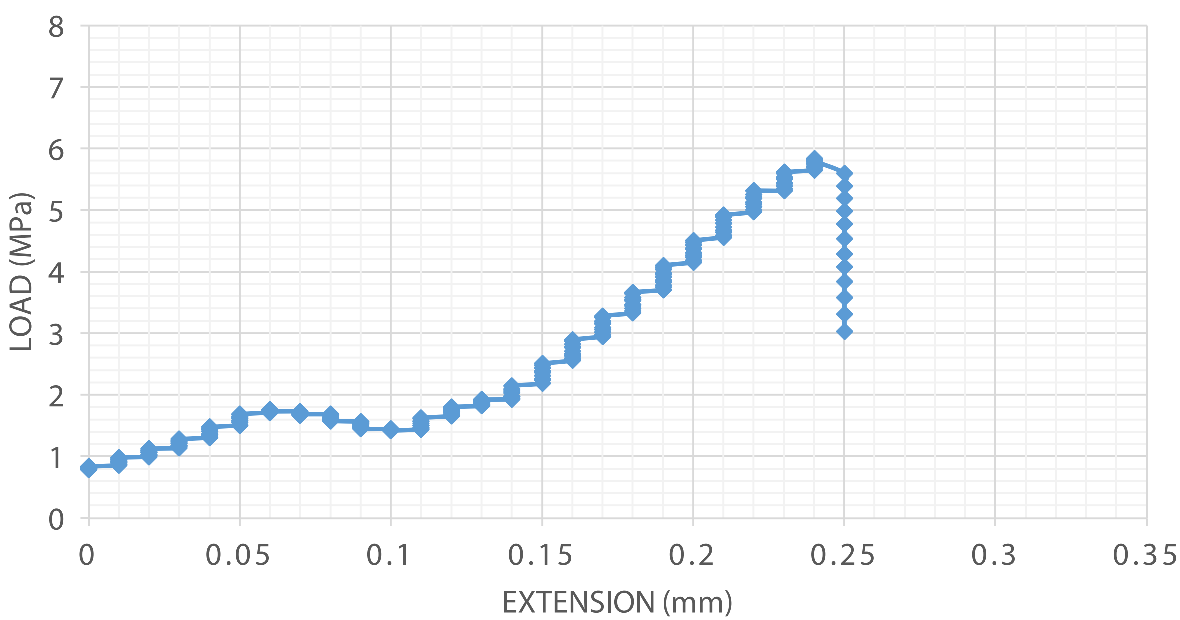

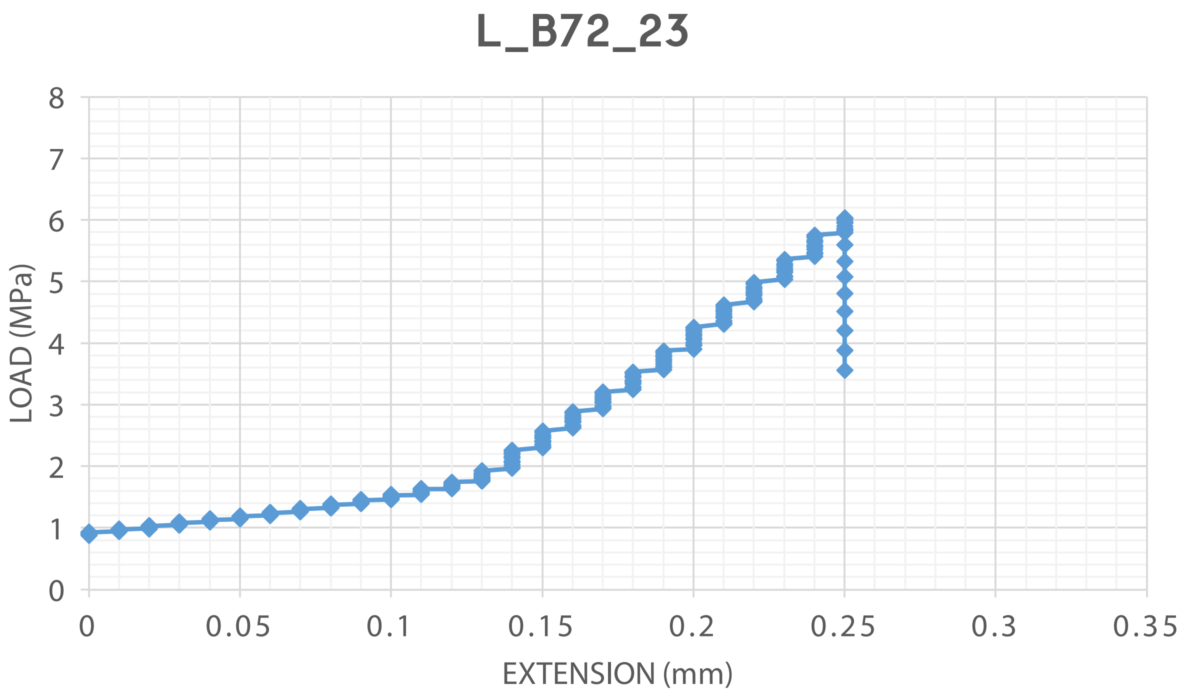
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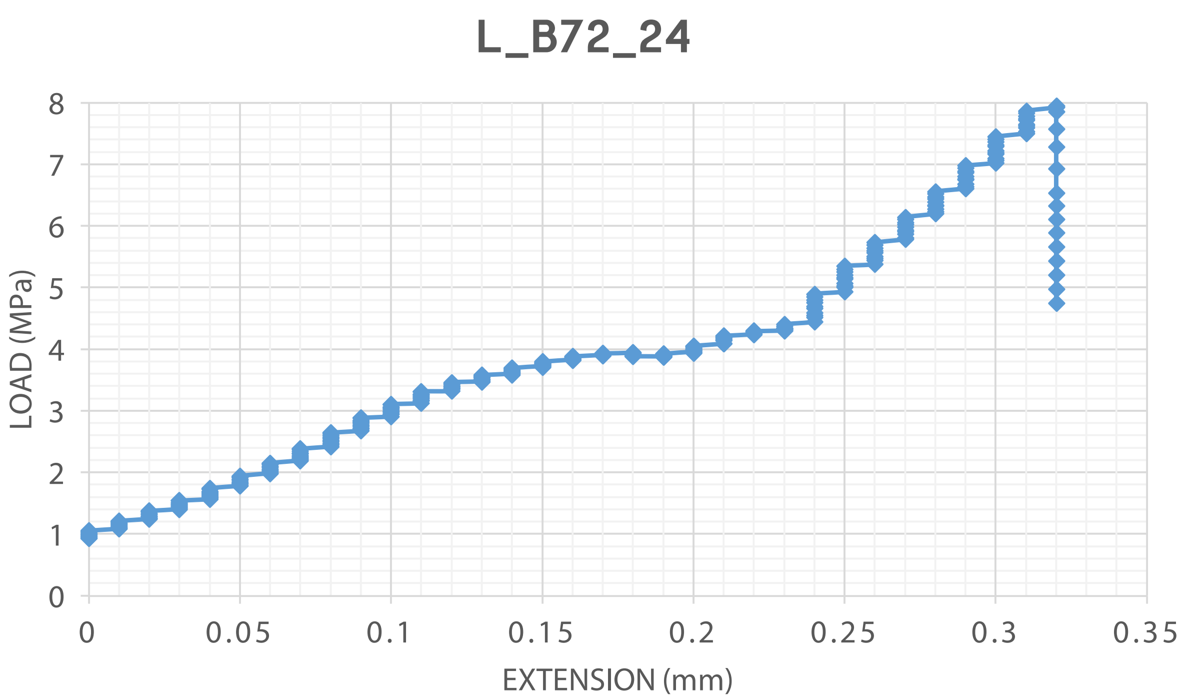
EXTENSION (mm)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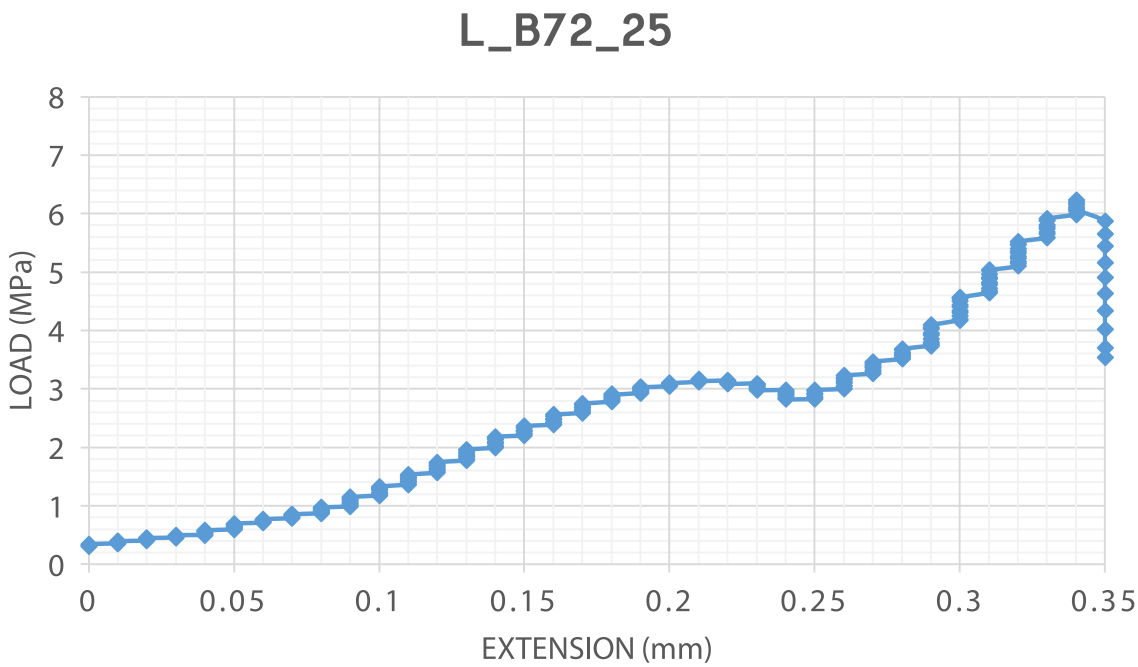
EXTENSION (mm)



L_B72_25
LOAD (MPa)
EXTENSION (mm)
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0.25
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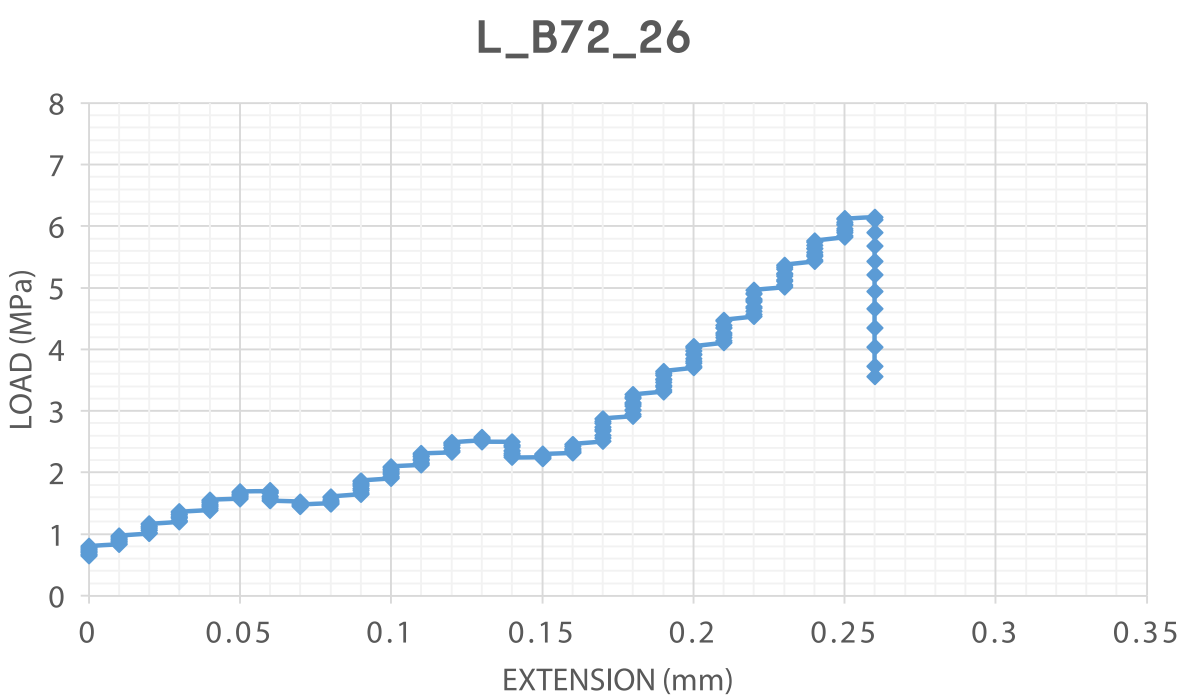
0.35



L_B72_26
LOAD (MPa)
EXTENSION (mm)
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0.15
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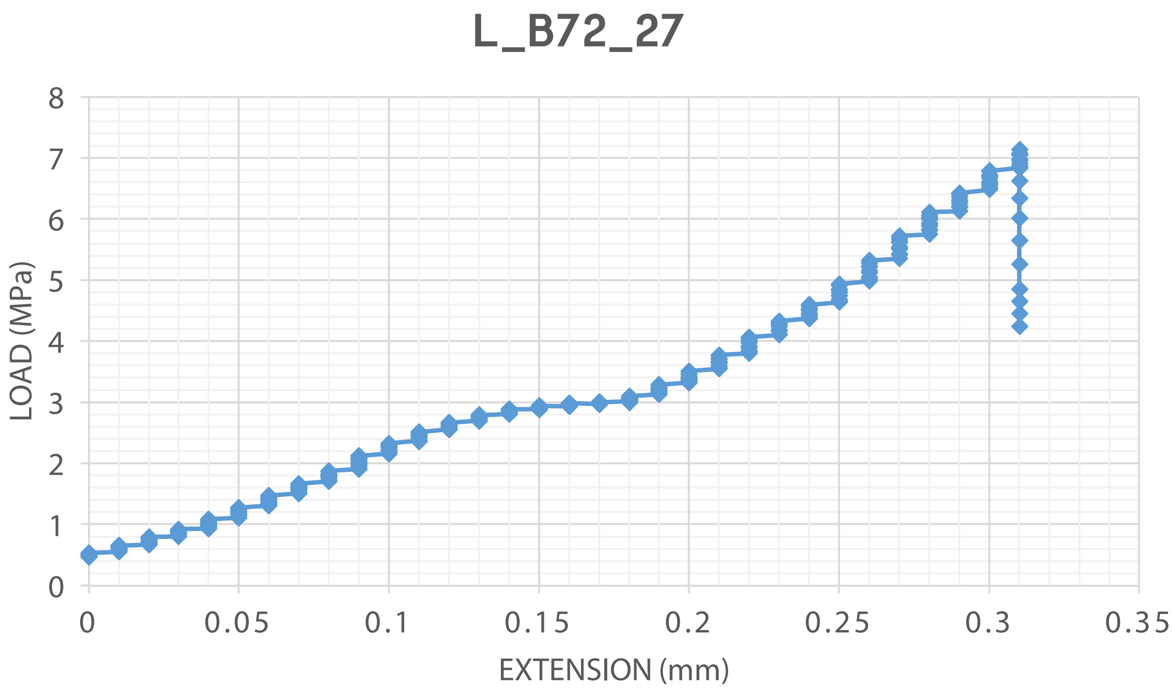
L_B72_27
LOAD (MPa)
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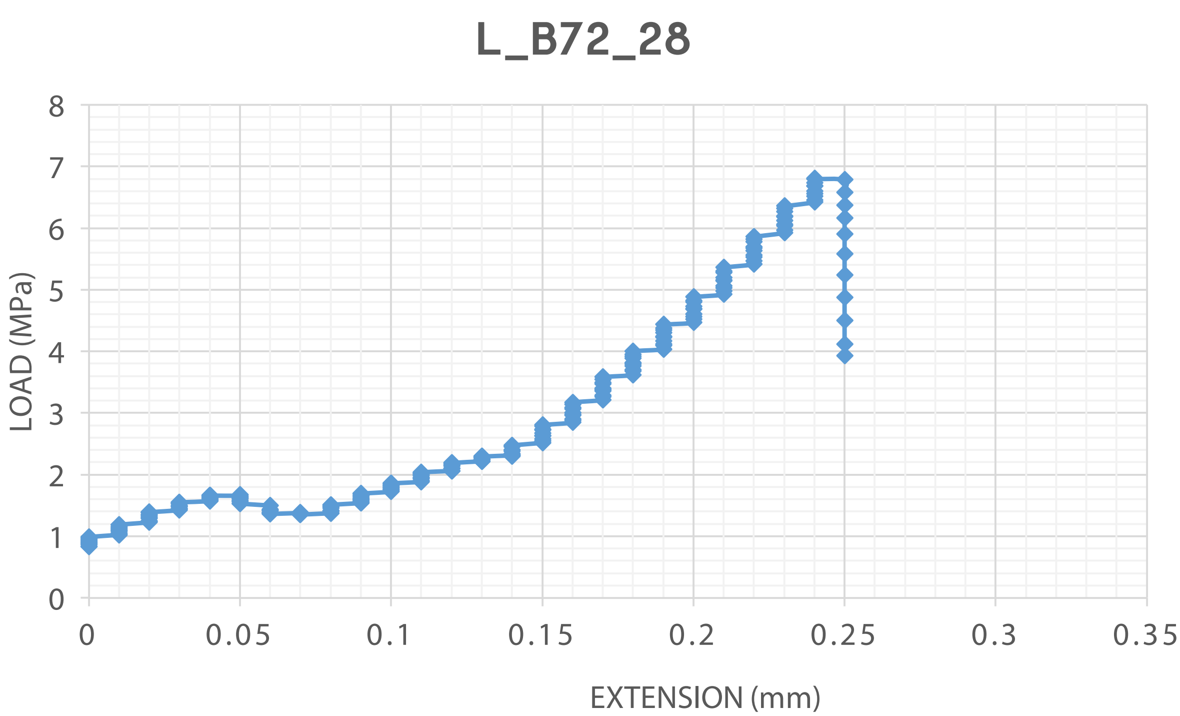
EXTENSION (mm)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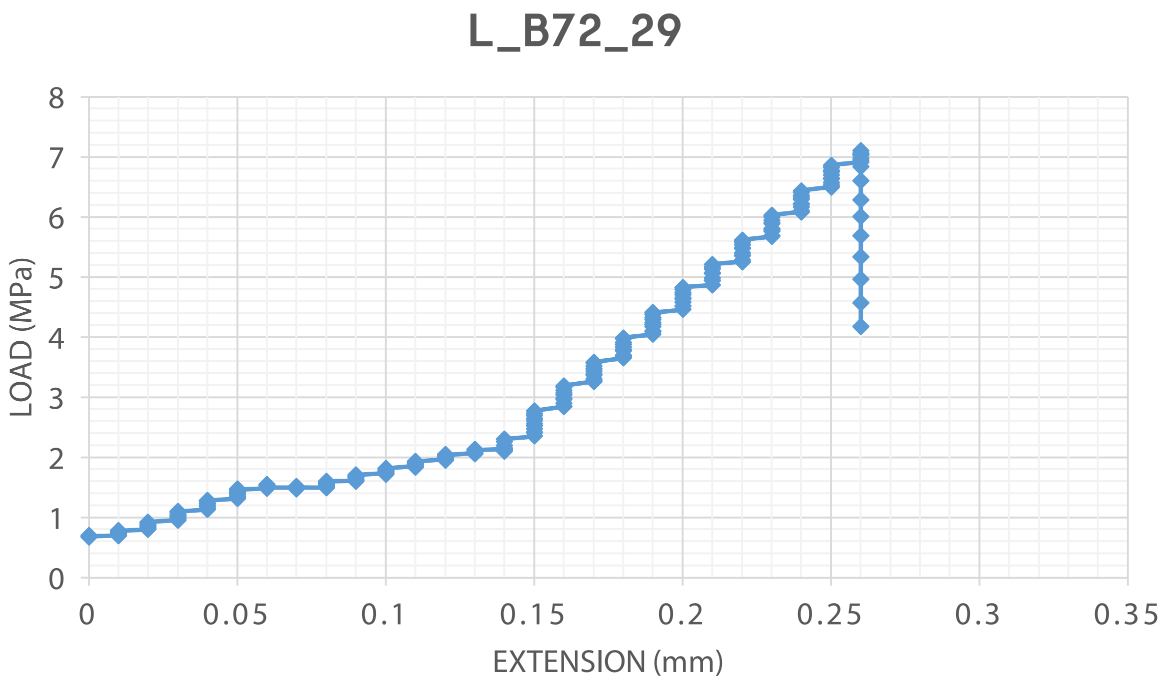
0.3
0.35
EXTENSION (mm)


L_B72_29
LOAD (MPa)
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3
4
5
6
7
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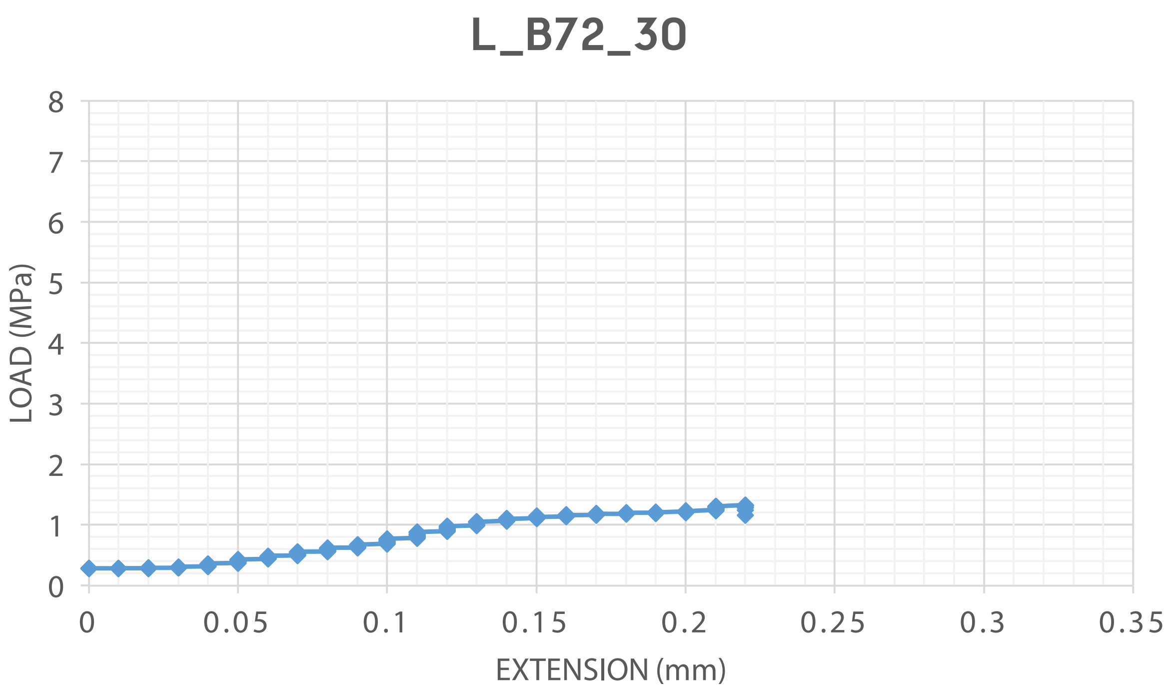
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B72_30
LOAD (MPa)
0
1
2
3
4
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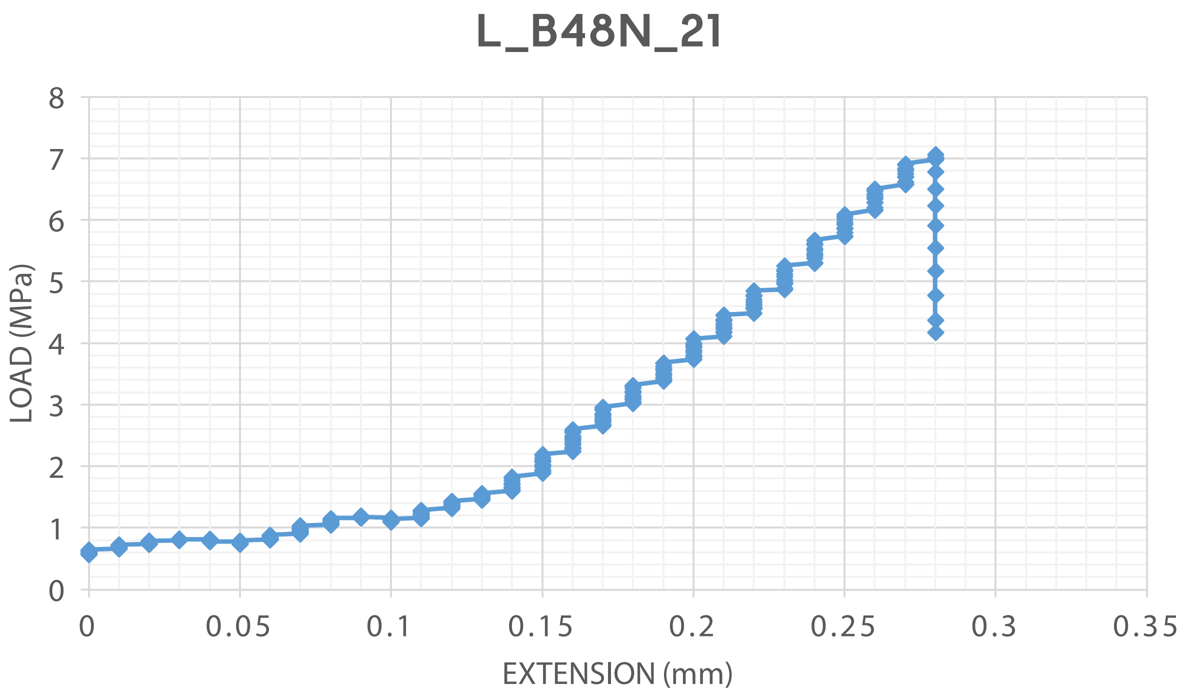
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B48N_21
LOAD (MPa)
EXTENSION (mm)
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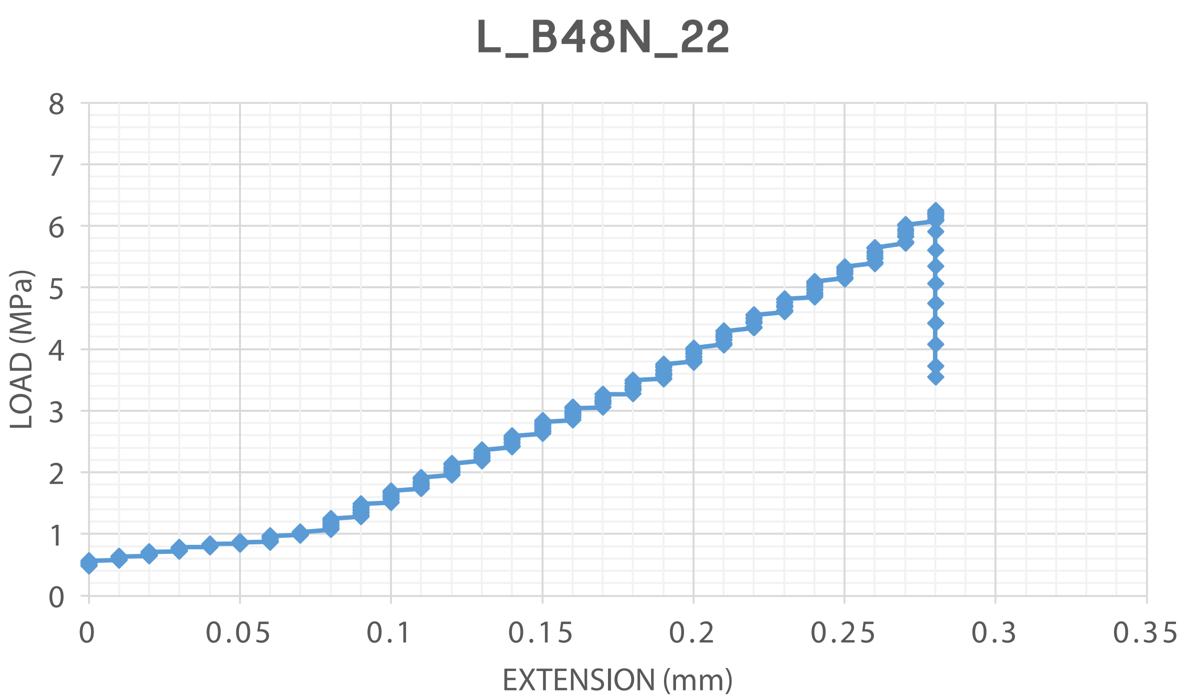
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B48N_22
LOAD (MPa)
EXTENSION (mm)
0
1
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3
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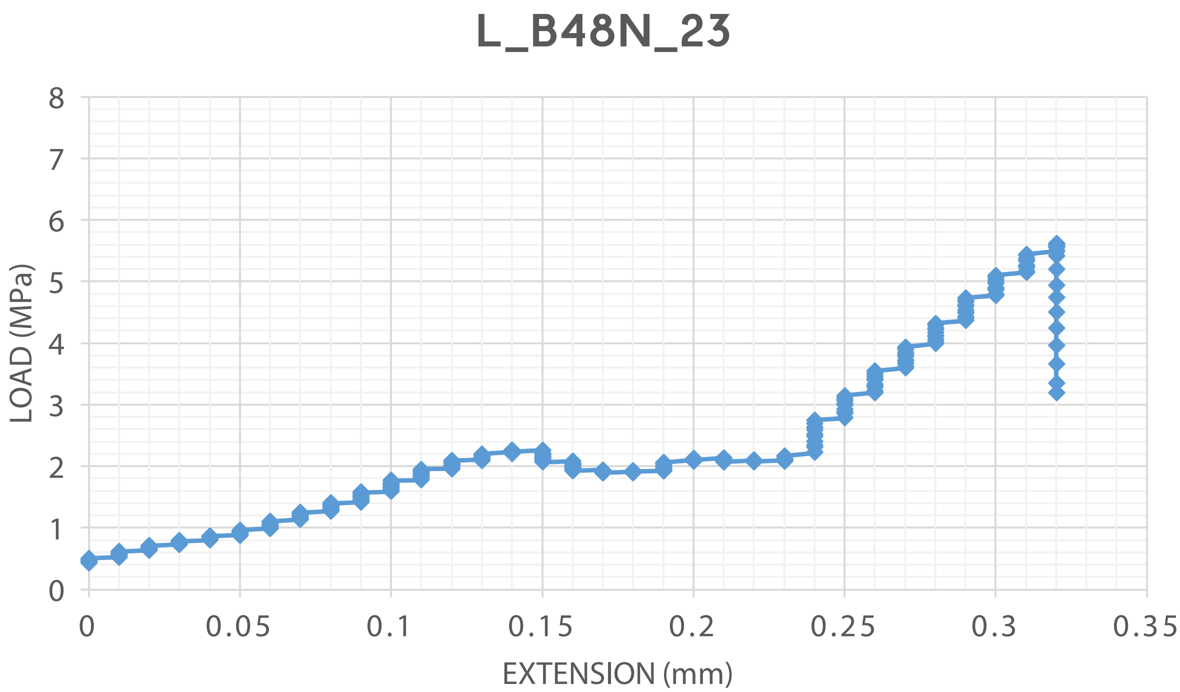
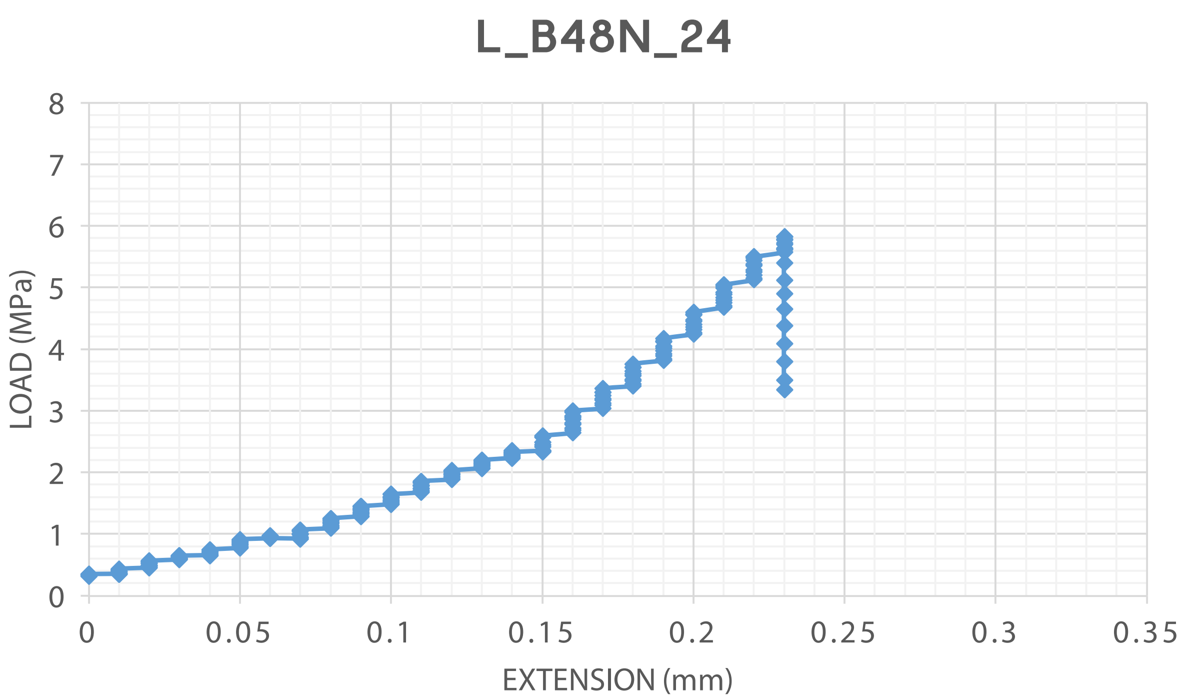
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B48N_23
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B48N_24
LOAD (MPa)
EXTENSION (mm)
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3
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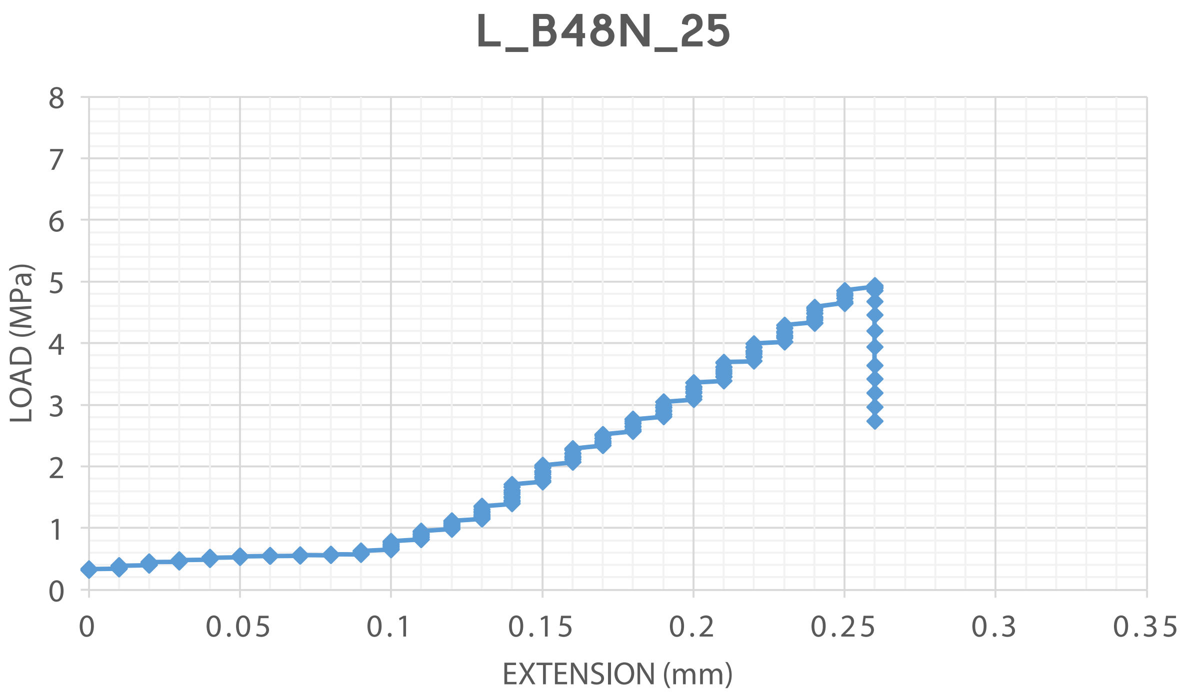
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B48N_25
LOAD (MPa)
EXTENSION (mm)
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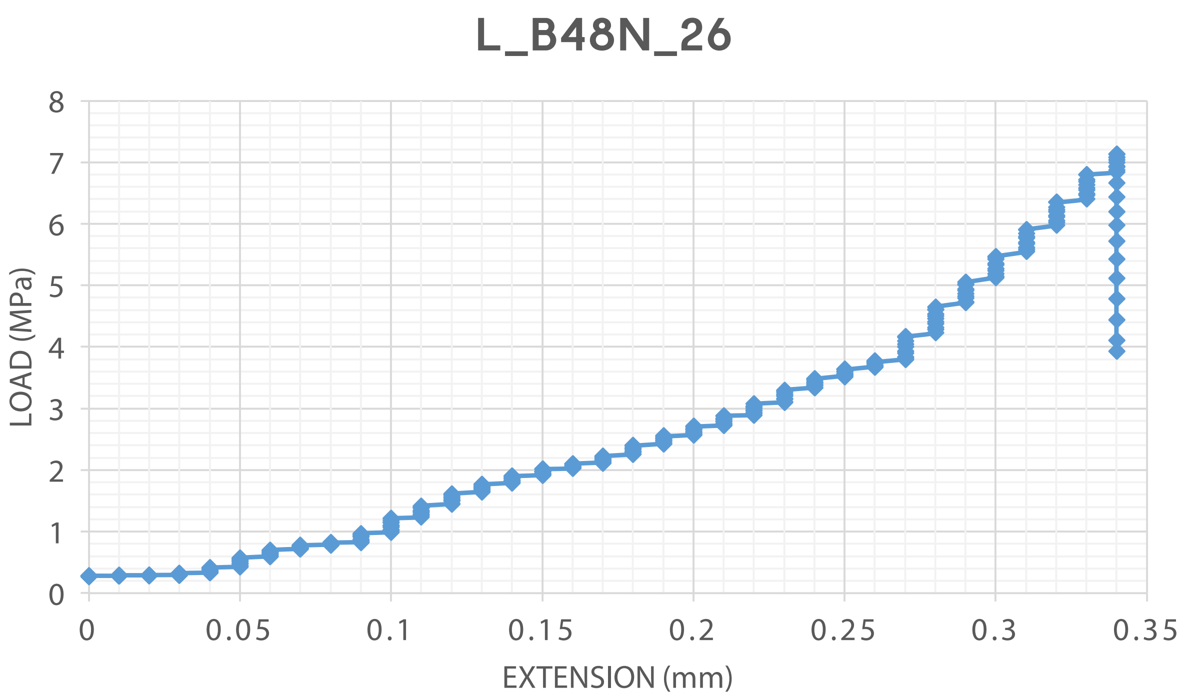
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B48N_26
LOAD (MPa)
EXTENSION (mm)
0
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3
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5
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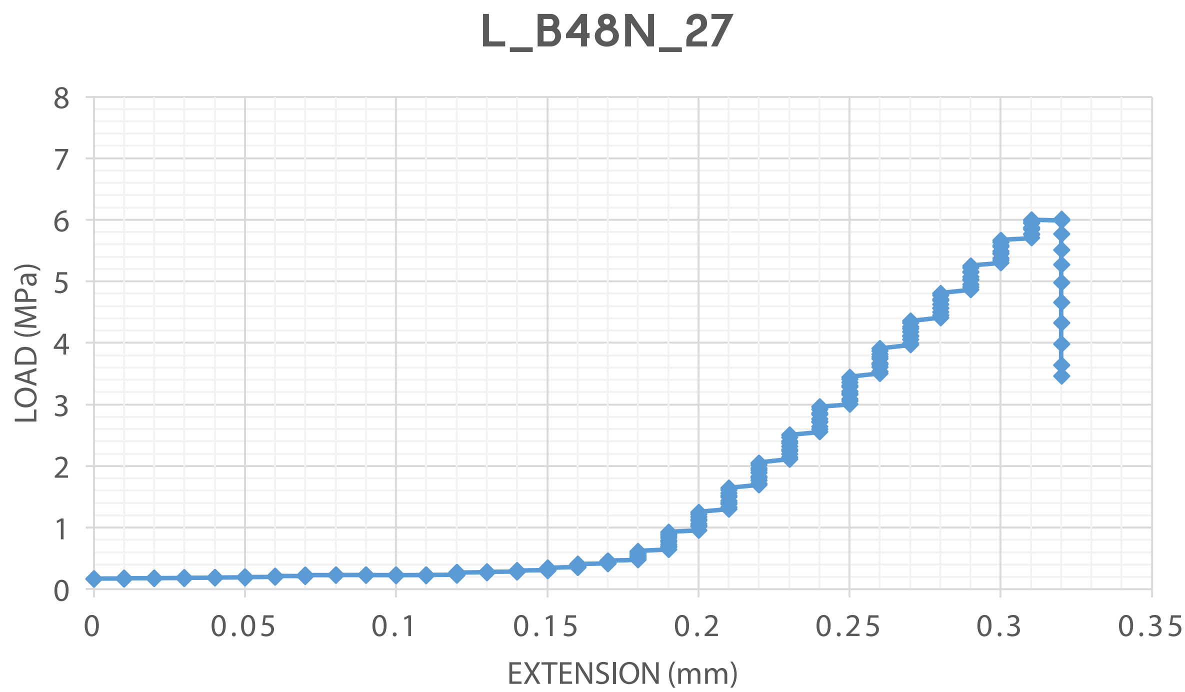
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B48N_27
LOAD (MPa)
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1
2
3
4
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6
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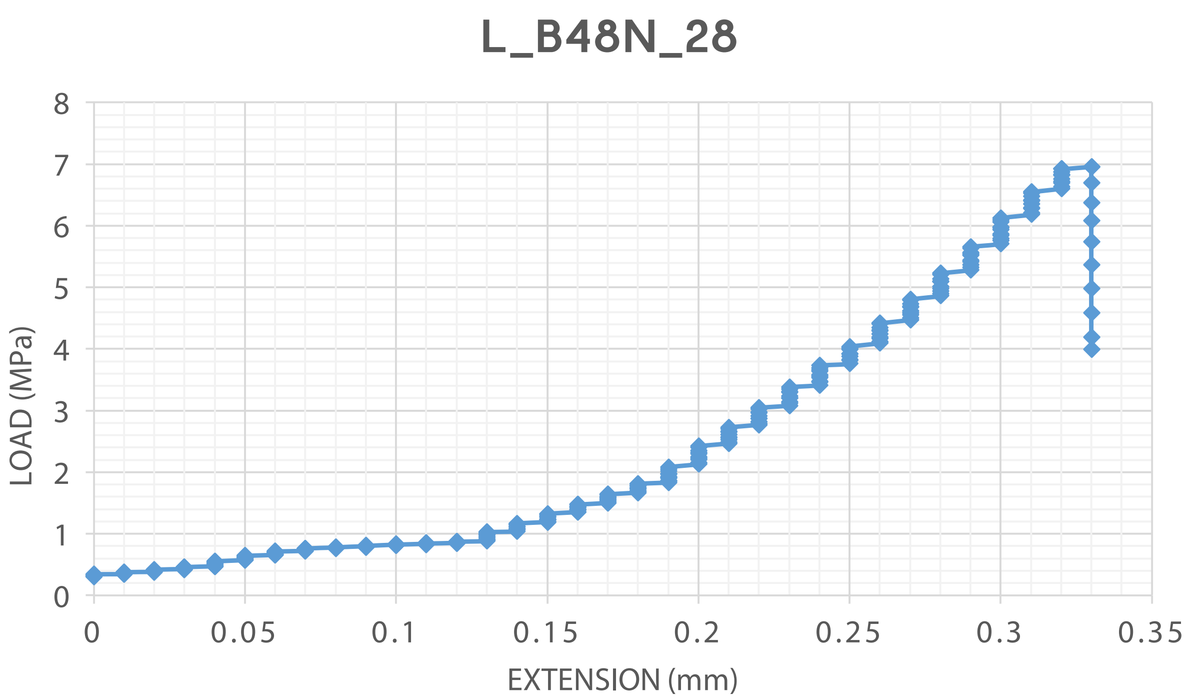
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B48N_28
LOAD (MPa)
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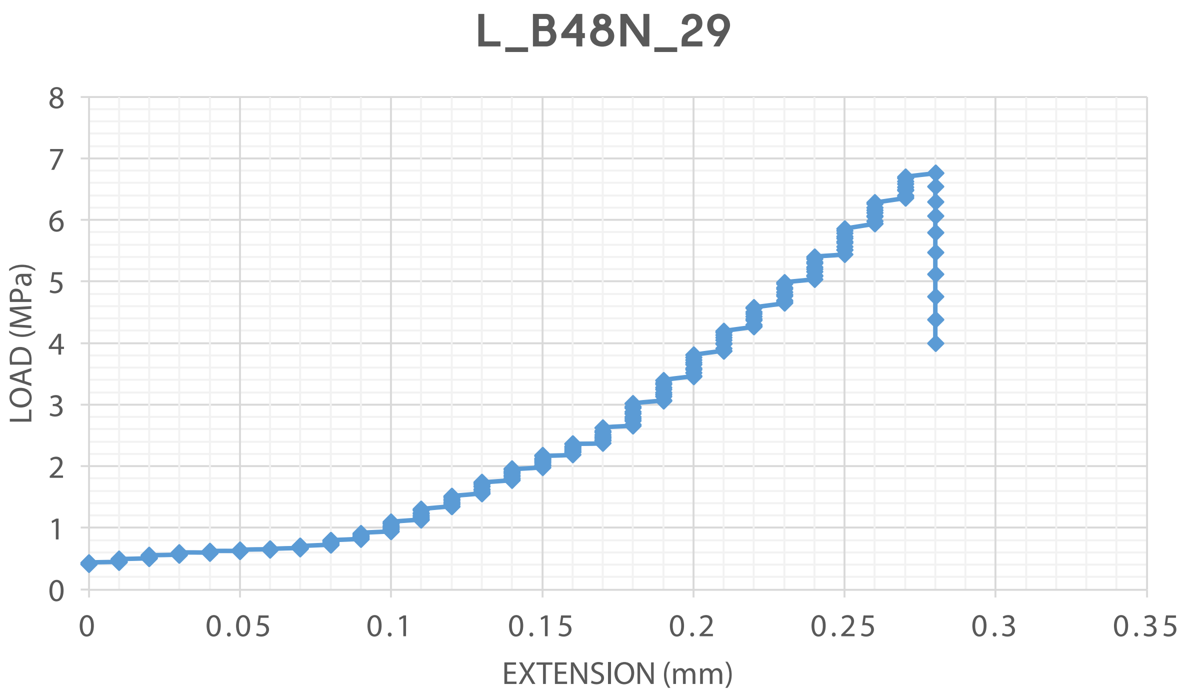
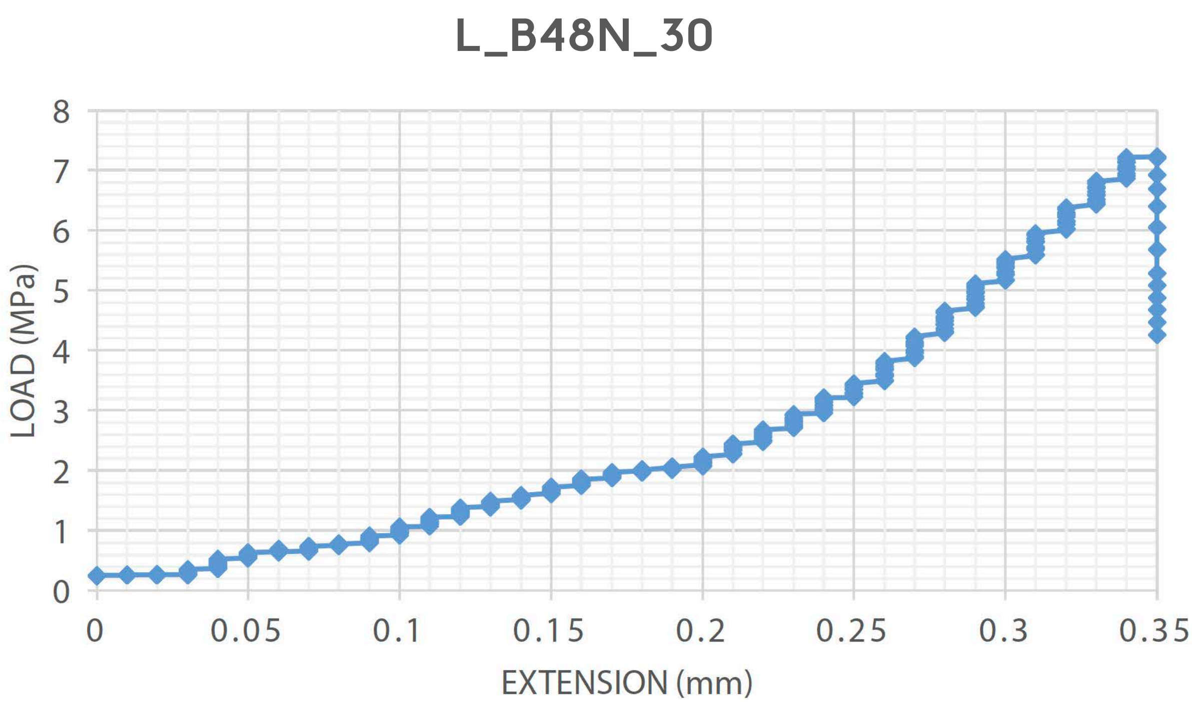
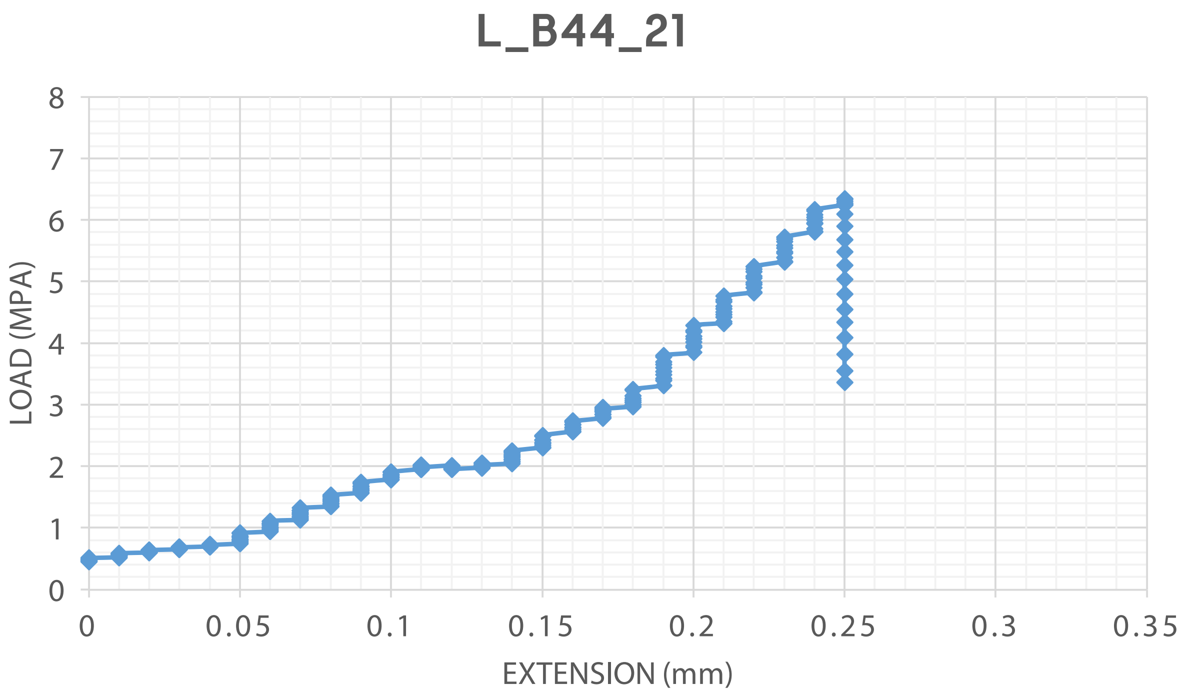
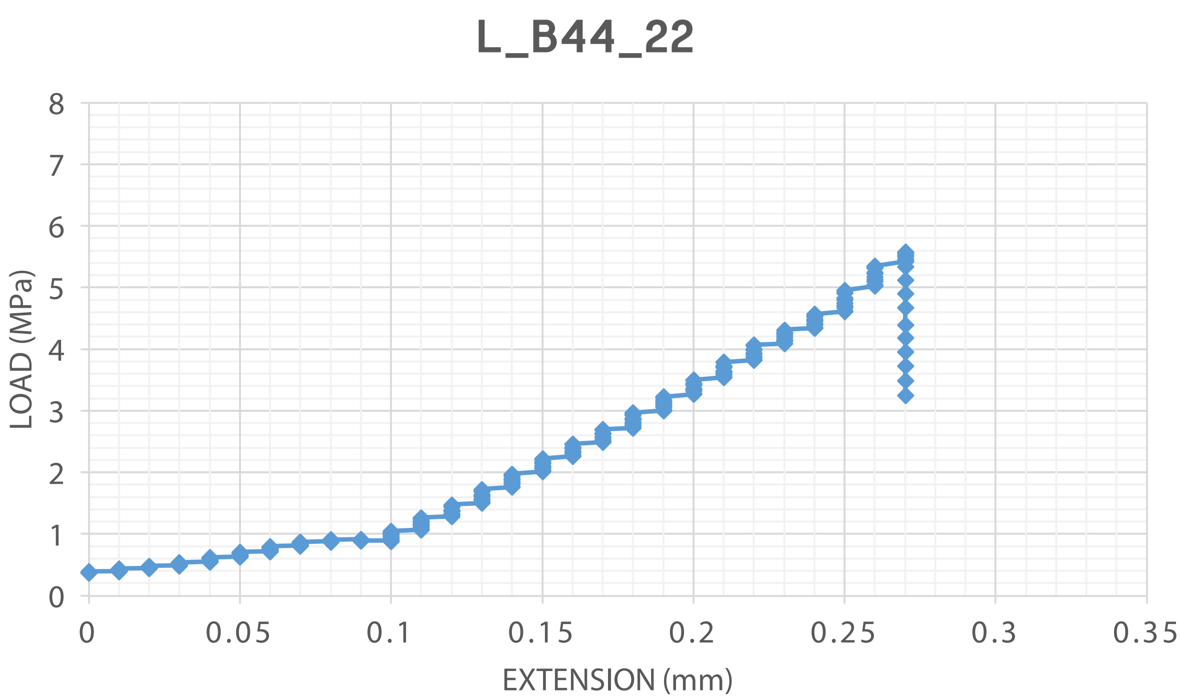
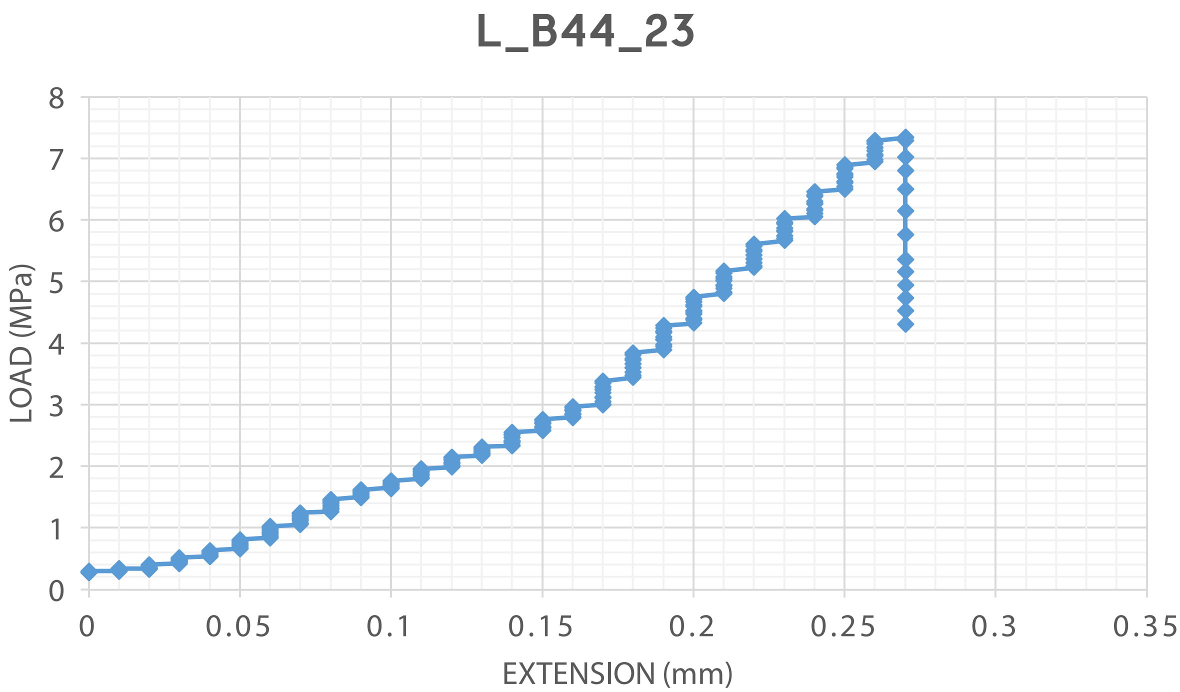
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B48N_29
LOAD (MPa)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)

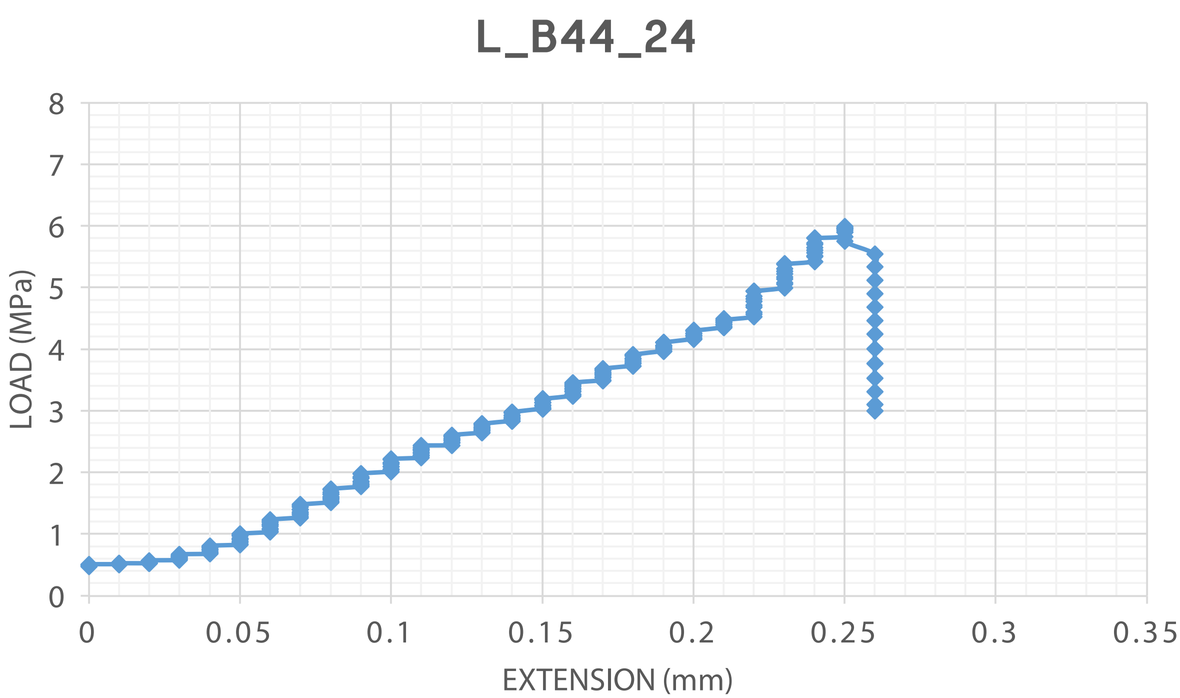

L_B48N_30
LOAD (MPa)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B44_21
LOAD (MPA)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B44_22
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35



L_B44_23
LOAD (MPa)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)



L_B44_24
LOAD (MPa)
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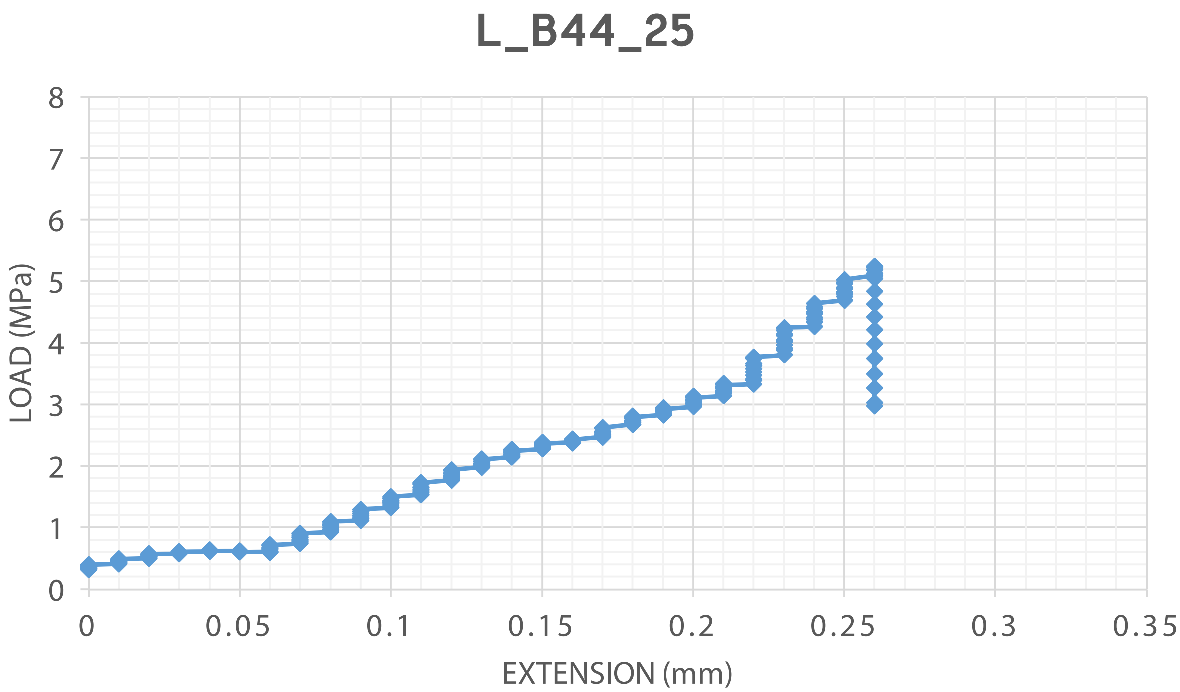
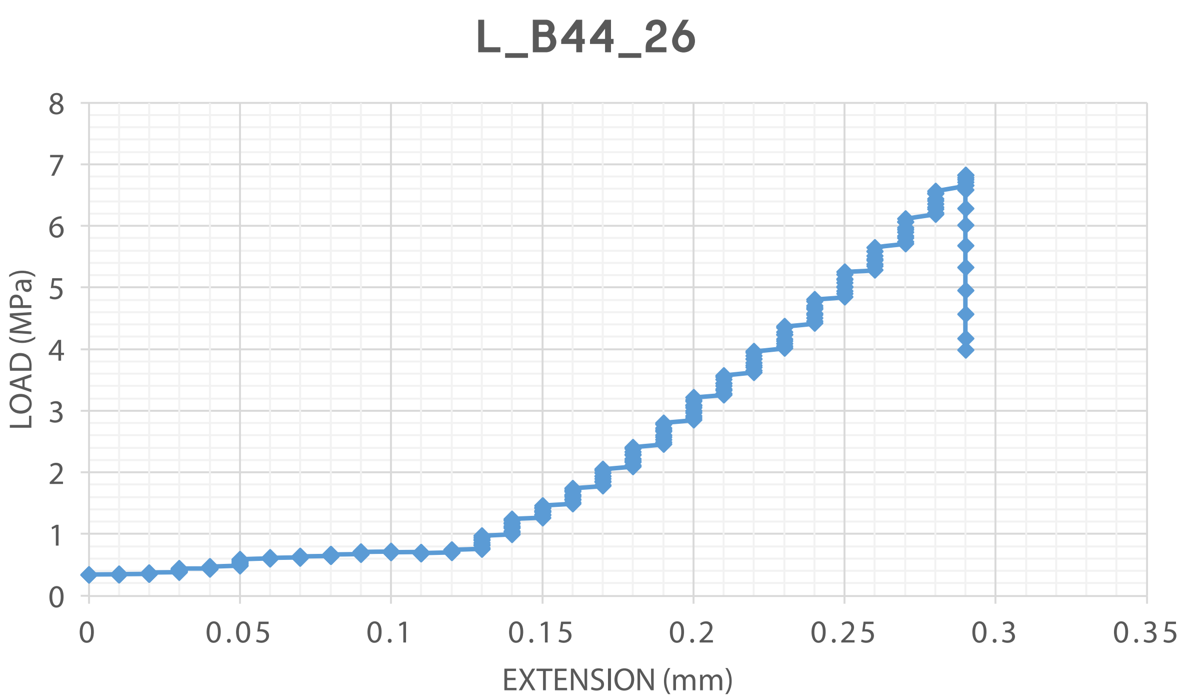
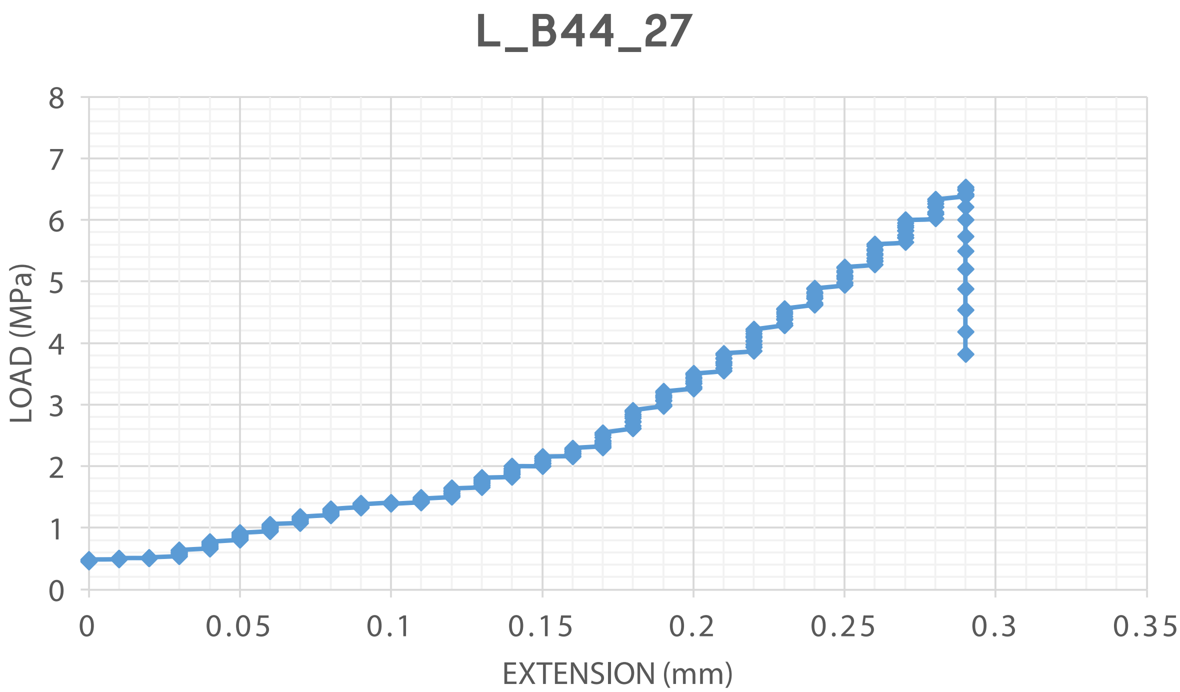
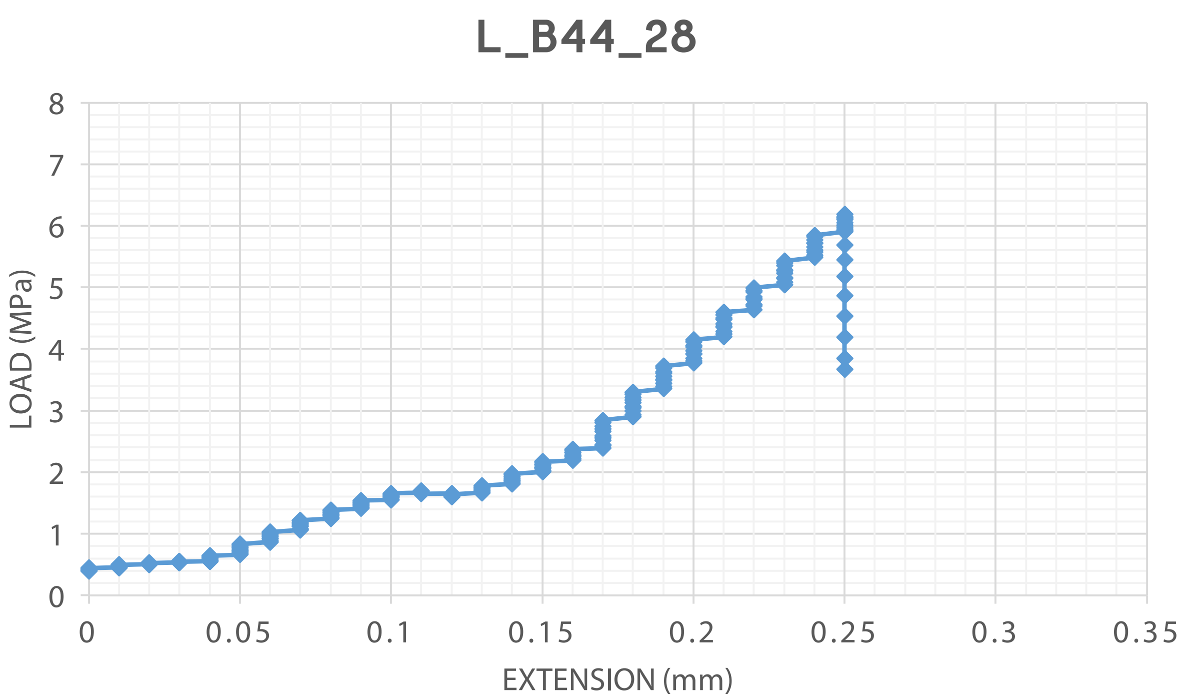
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B44_25
LOAD (MPa)
8
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B44_26
LOAD (MPa)
8
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_B44_27
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B44_28
LOAD (MPa)
EXTENSION (mm)
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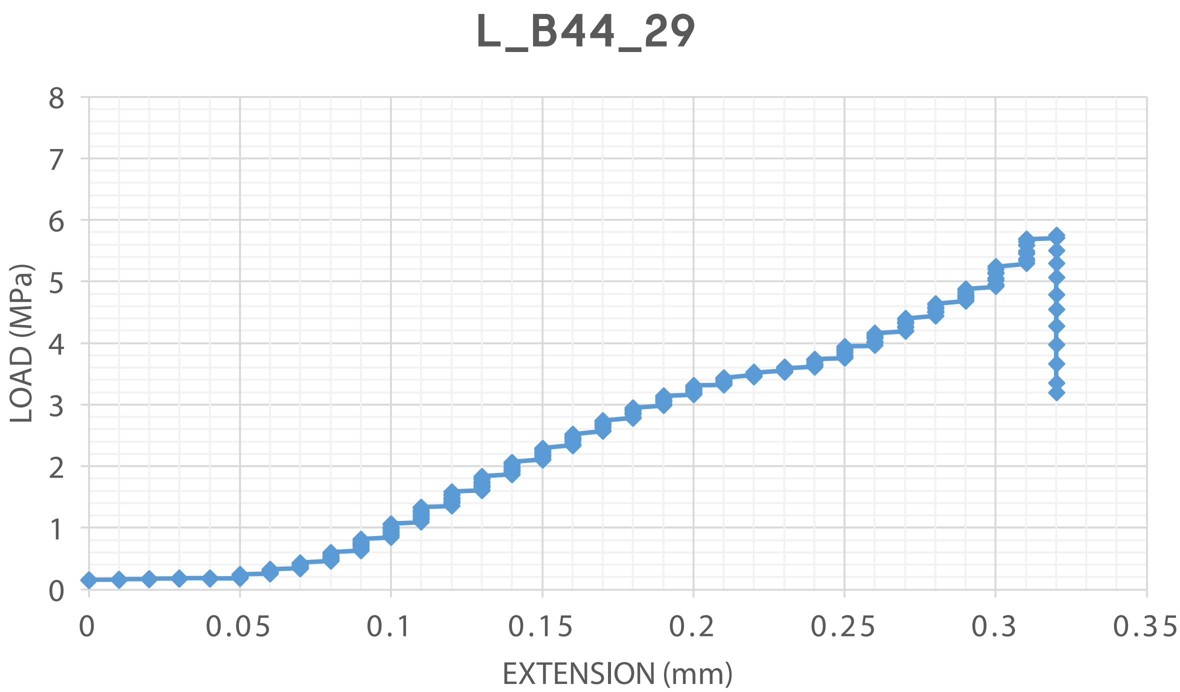
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B44_29
LOAD (MPa)
EXTENSION (mm)
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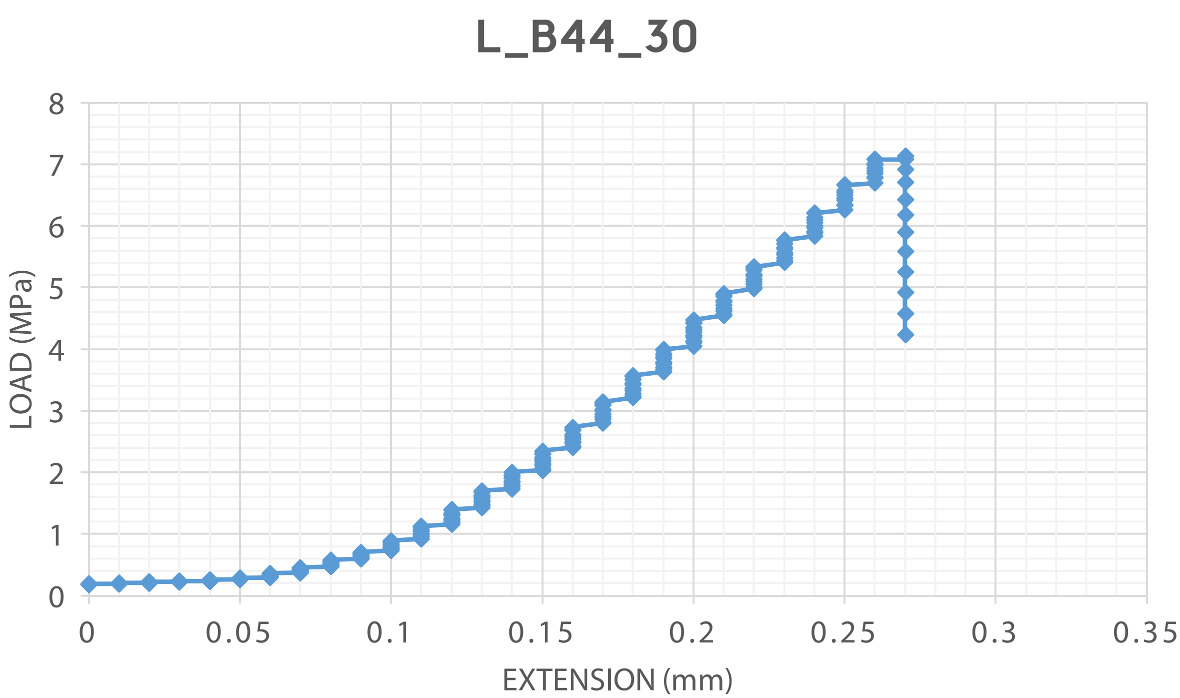
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_B44_30
LOAD (MPa)
EXTENSION (mm)
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1
2
3
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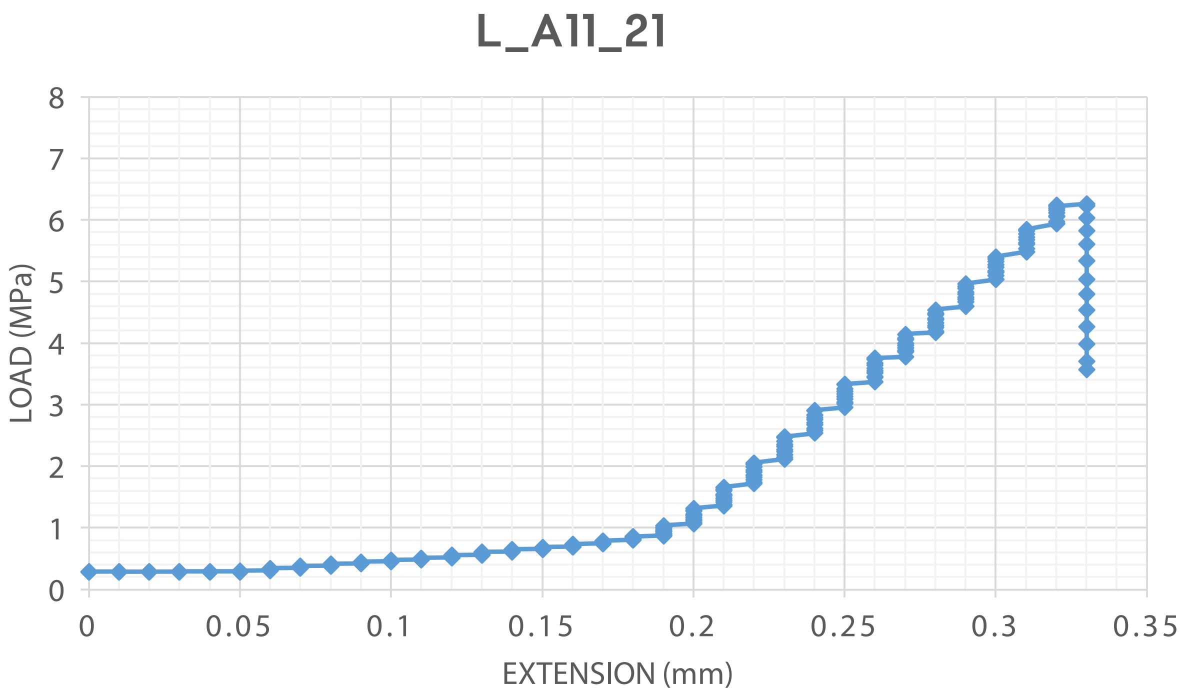
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_A11_21
LOAD (MPa)
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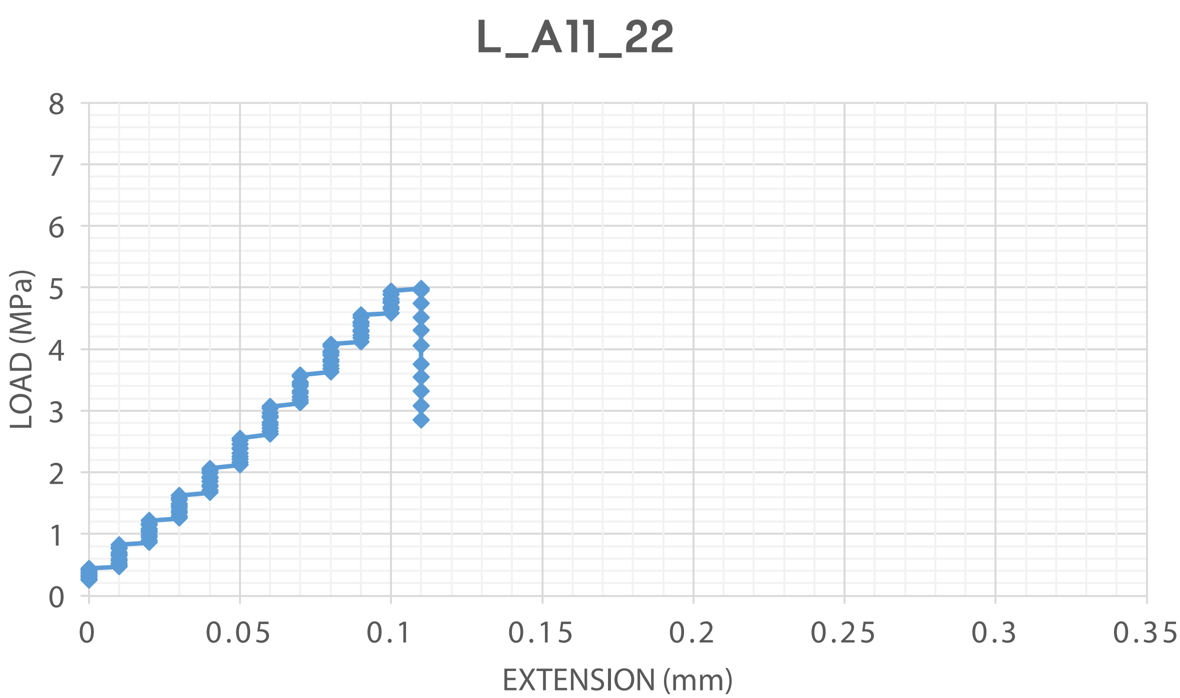
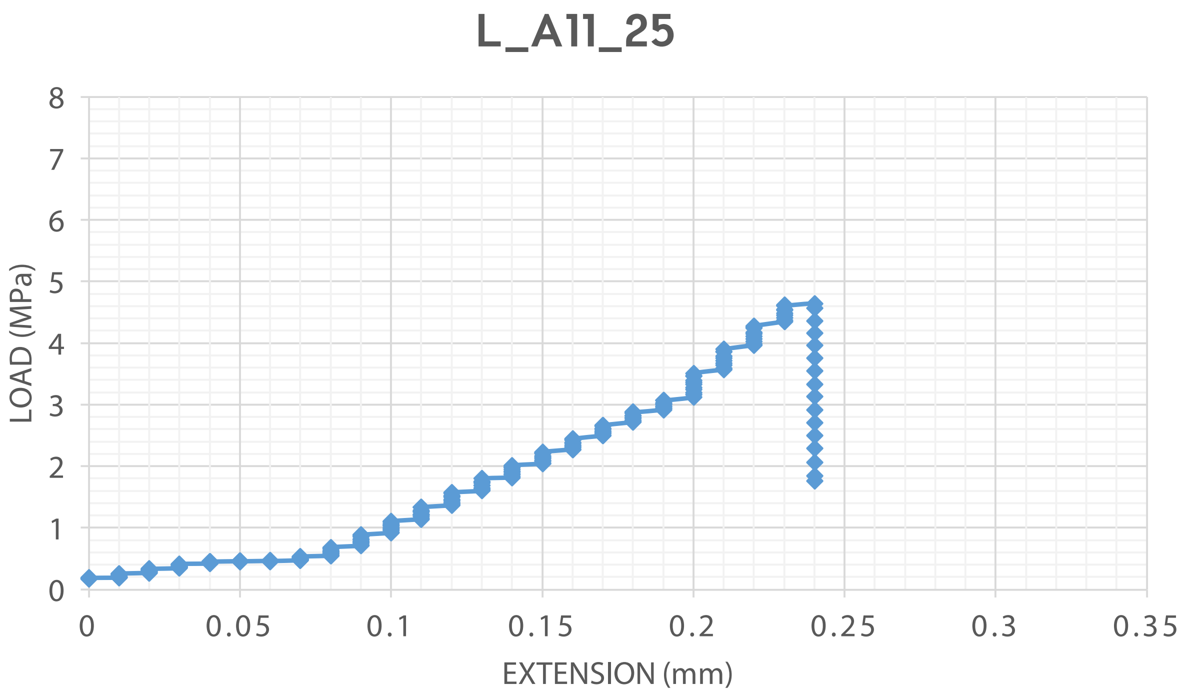
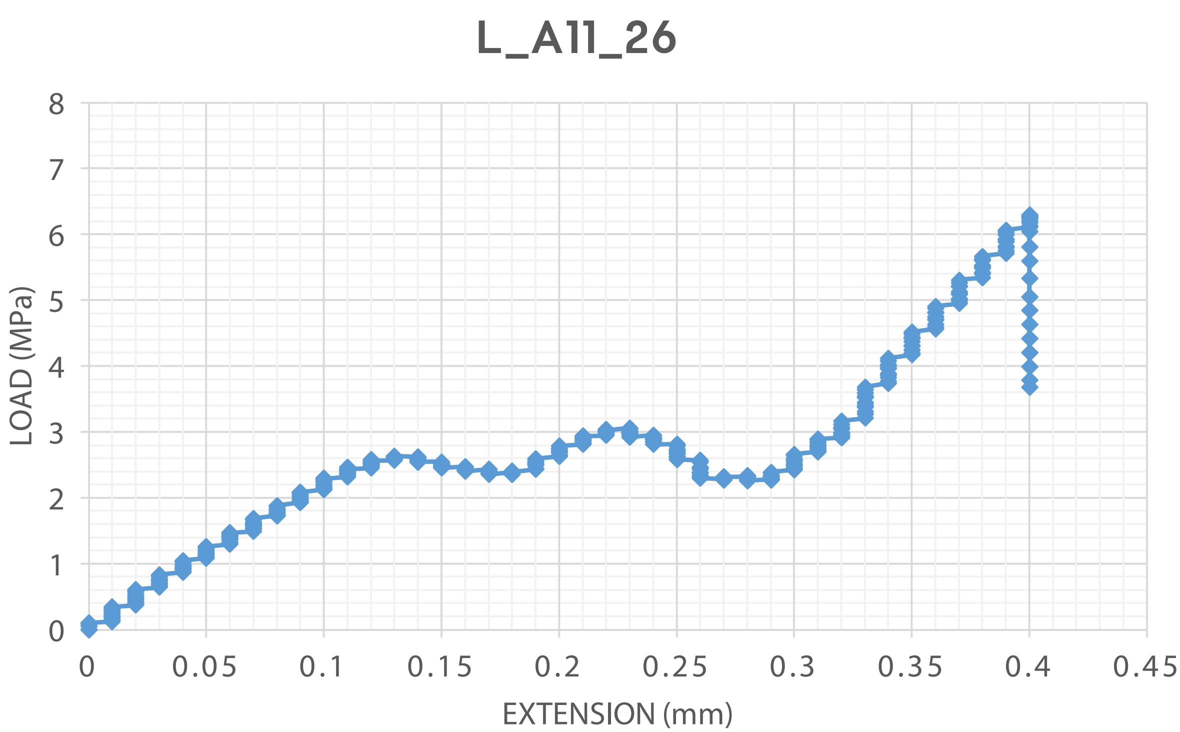
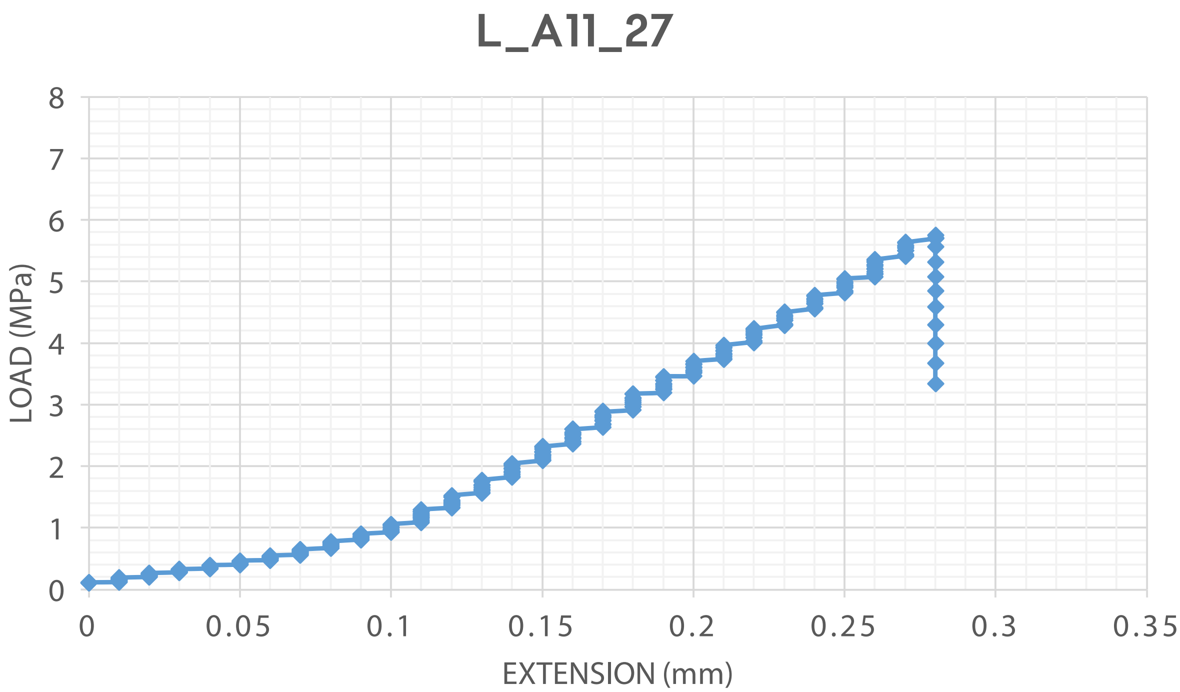
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_A11_22
LOAD (MPa)
8
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)





*scale different for A_11_26


L_A11_27
LOAD (MPa)
8
7
6
5
4
3
2
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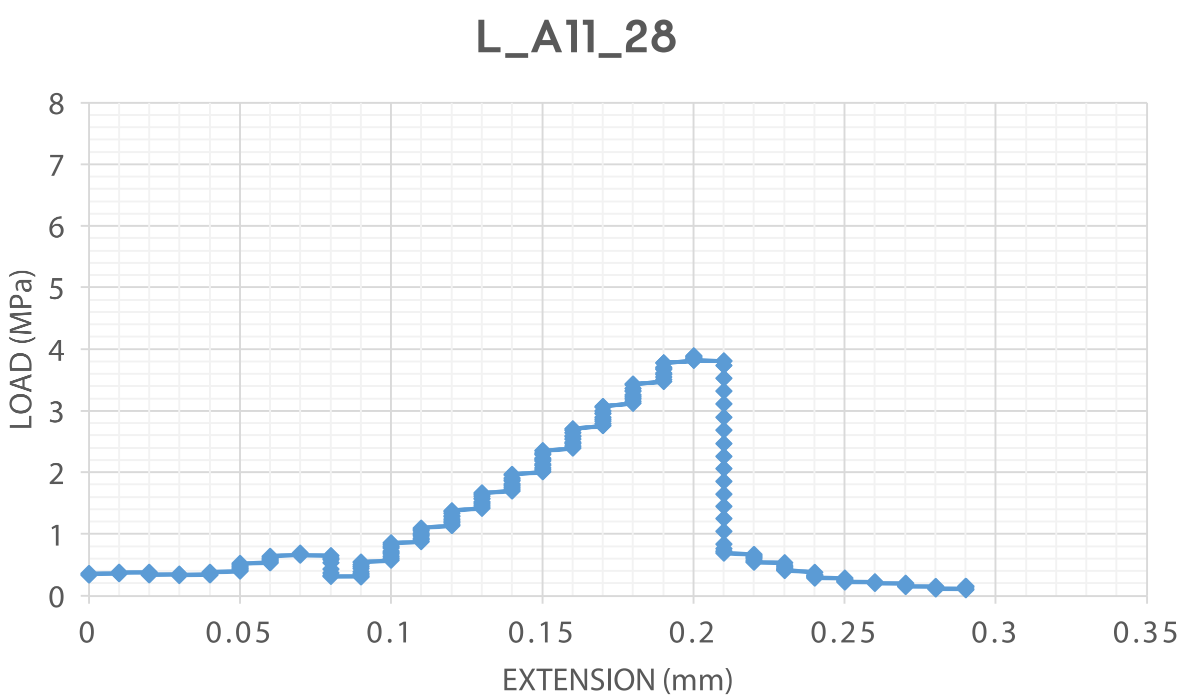
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_A11_28
LOAD (MPa)
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7
6
5
4
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2
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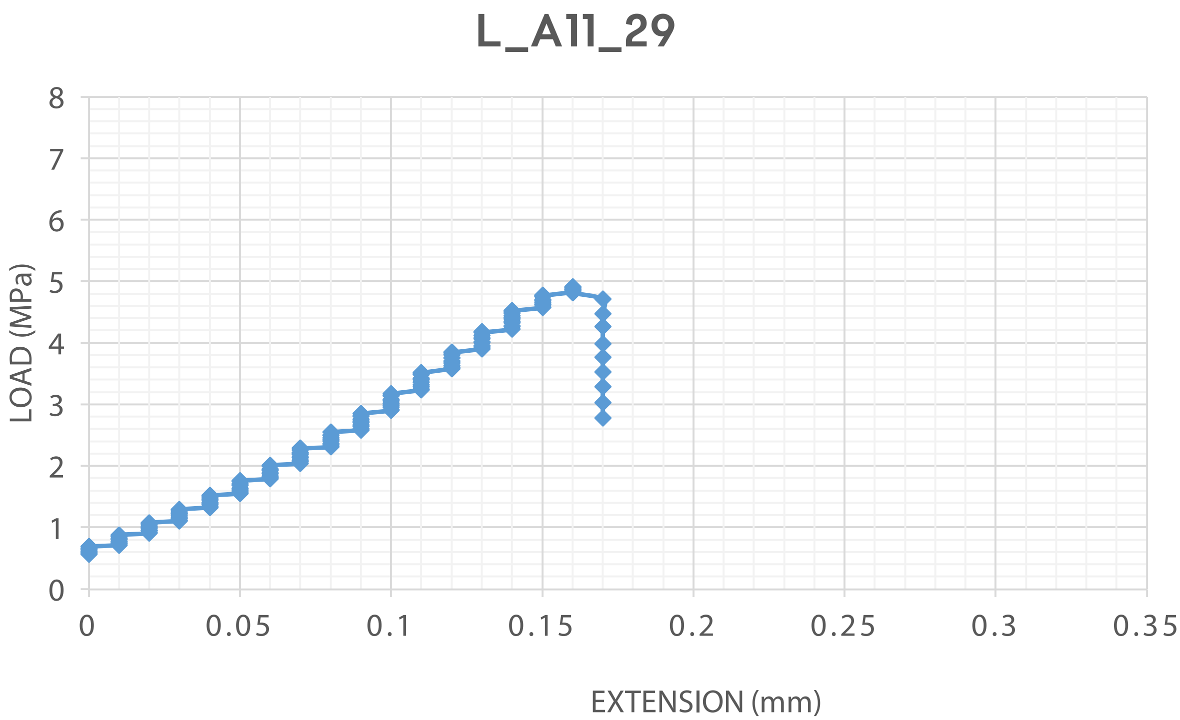
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_A11_29
LOAD (MPa)
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7
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5
4
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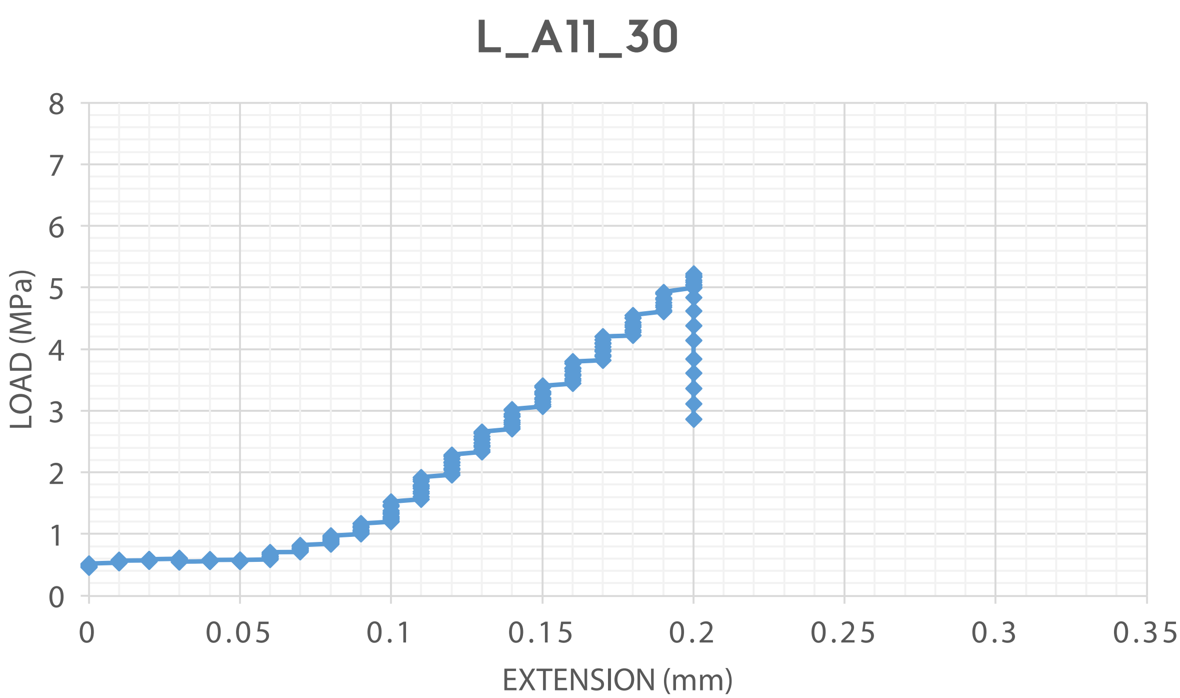
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_A11_30
LOAD (MPa)
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5
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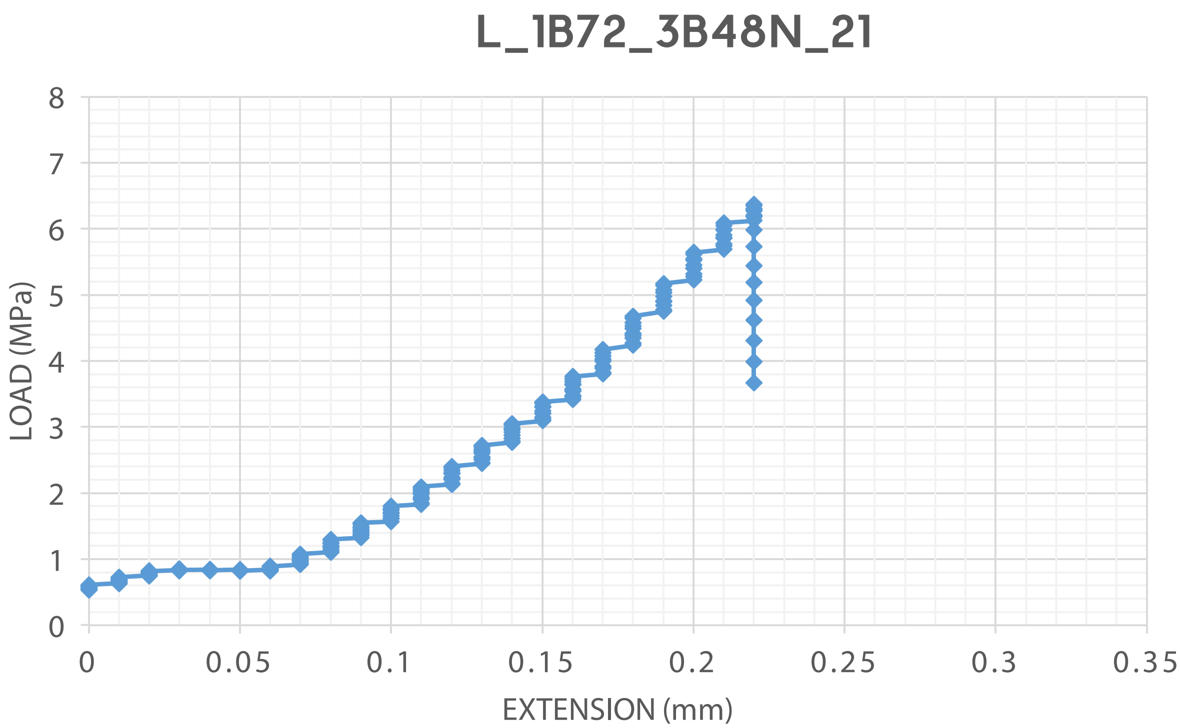
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_1B72_3B48N_21
LOAD (MPa)
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1
2
3
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6
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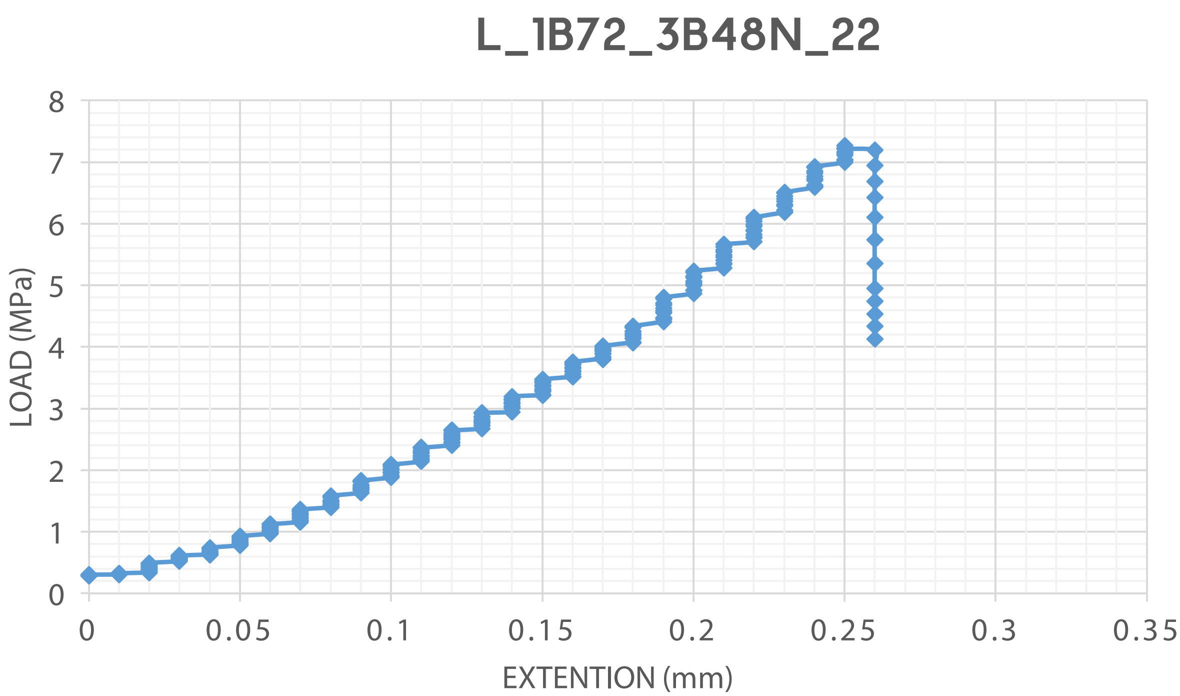
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_1B72_3B48N_22
LOAD (MPa)
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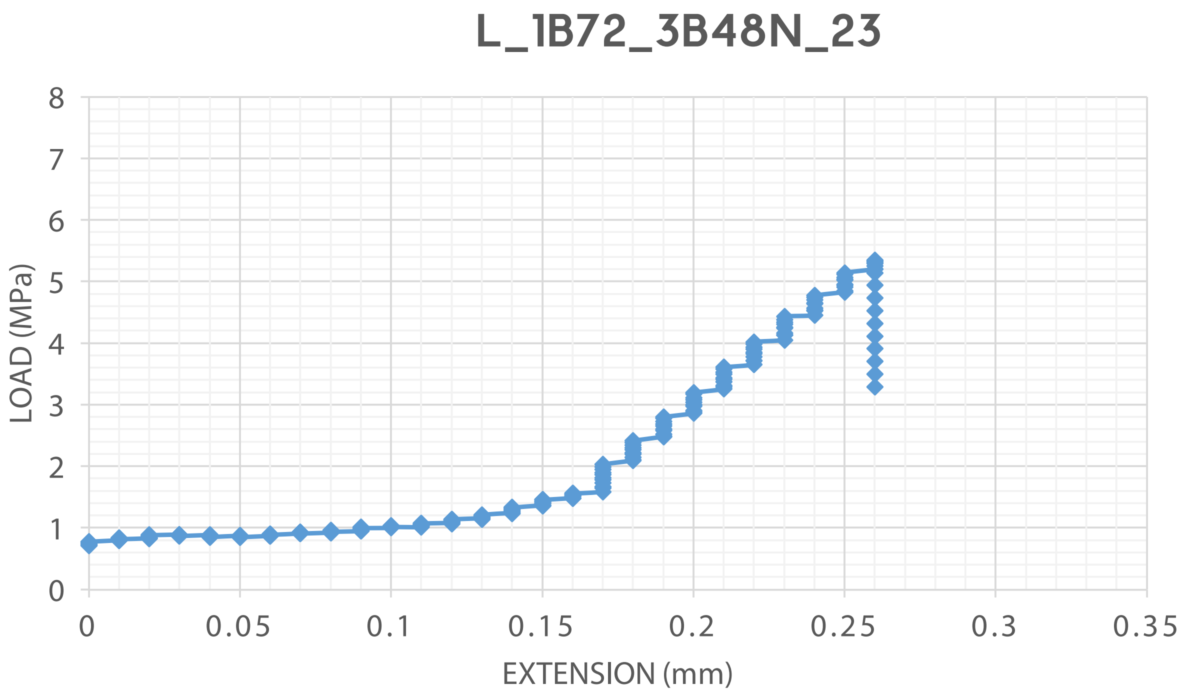
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENTION (mm)


L_1B72_3B48N_23
LOAD (MPa)
EXTENSION (mm)
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3
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5
6
7
8
0
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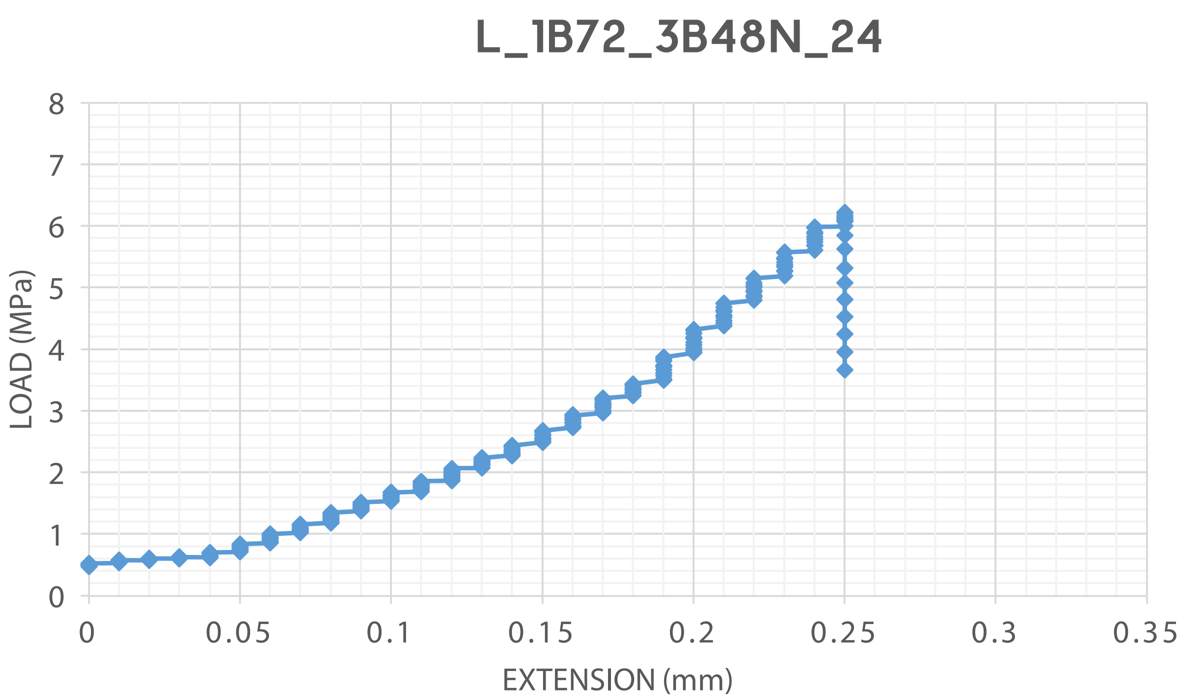
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_24
LOAD (MPa)
EXTENSION (mm)
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5
6
7
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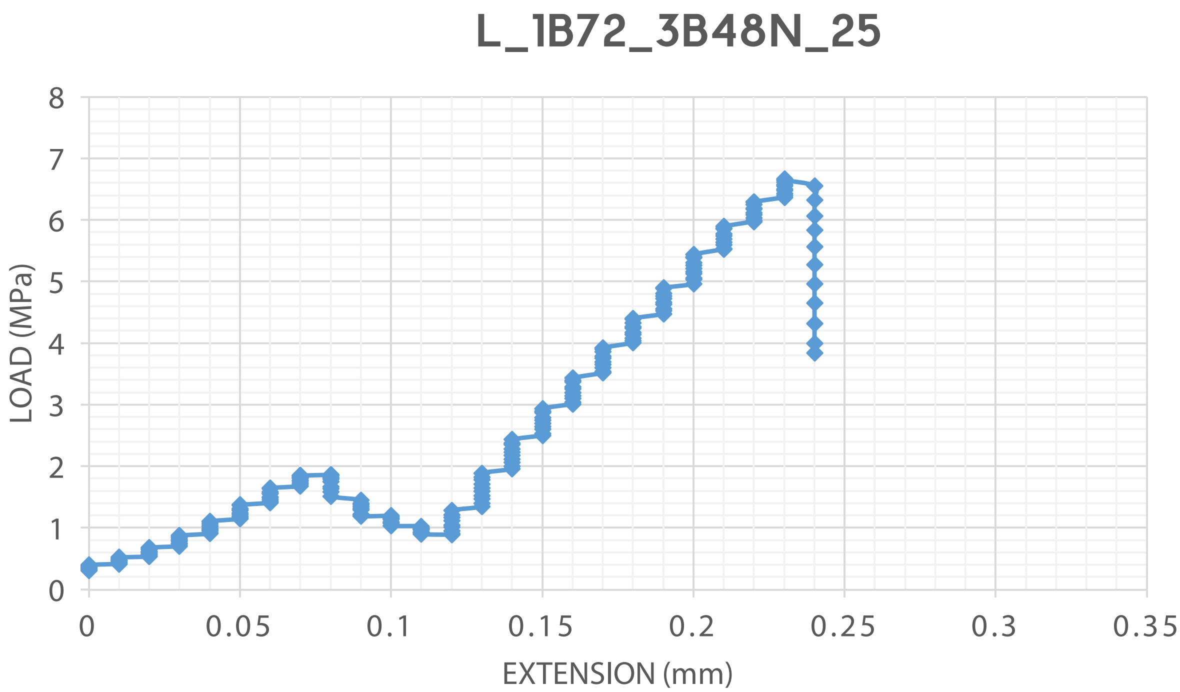
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_25
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
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6
7
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0.05
0.1
0.15
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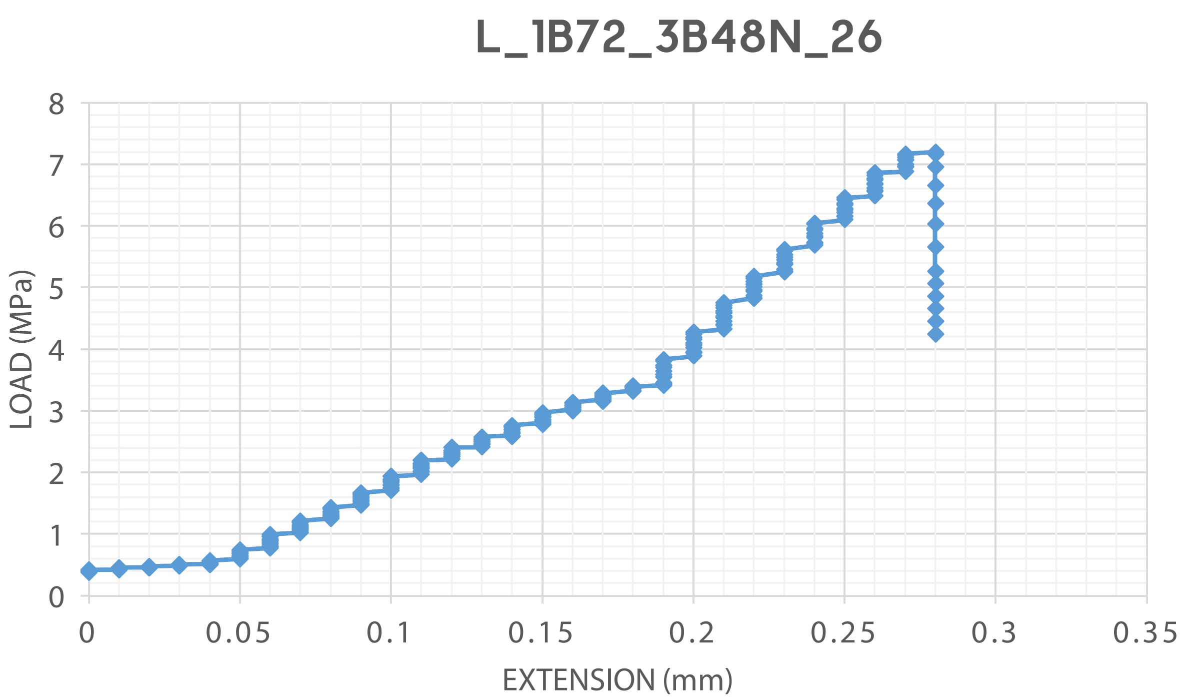
L_1B72_3B48N_26
LOAD (MPa)
EXTENSION (mm)
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3
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5
6
7
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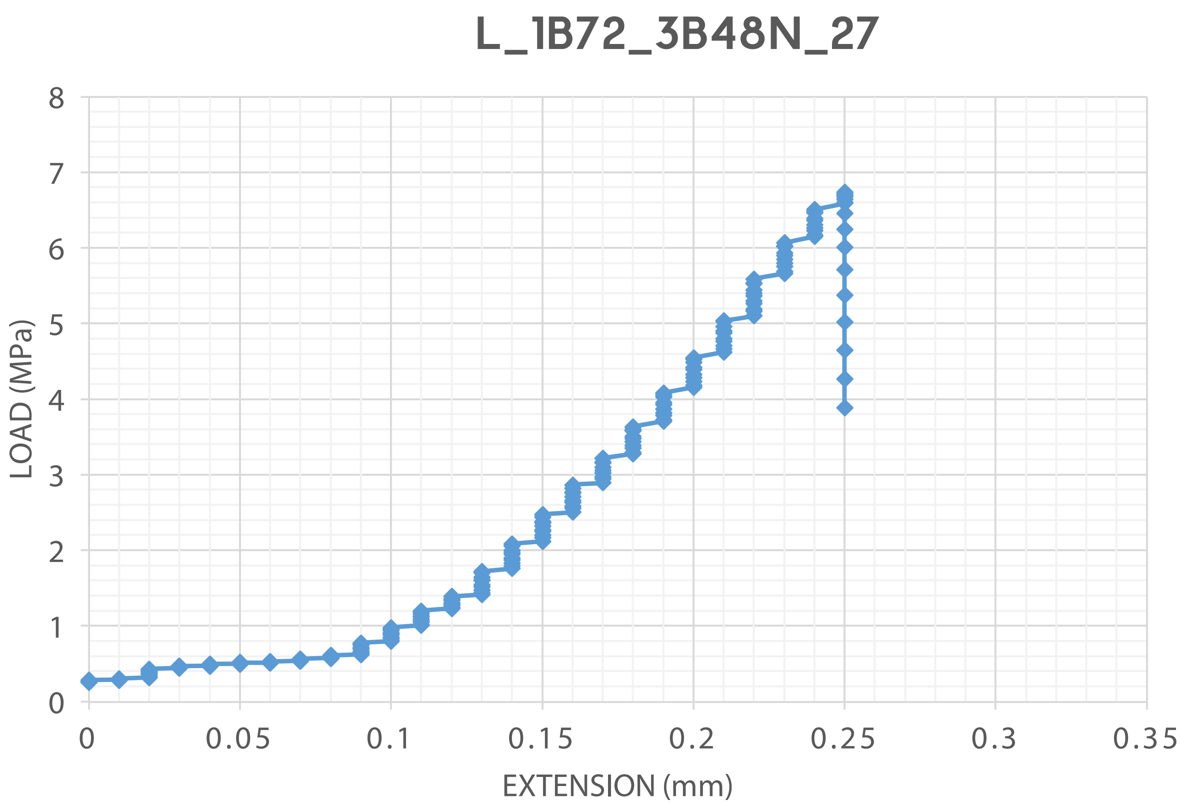
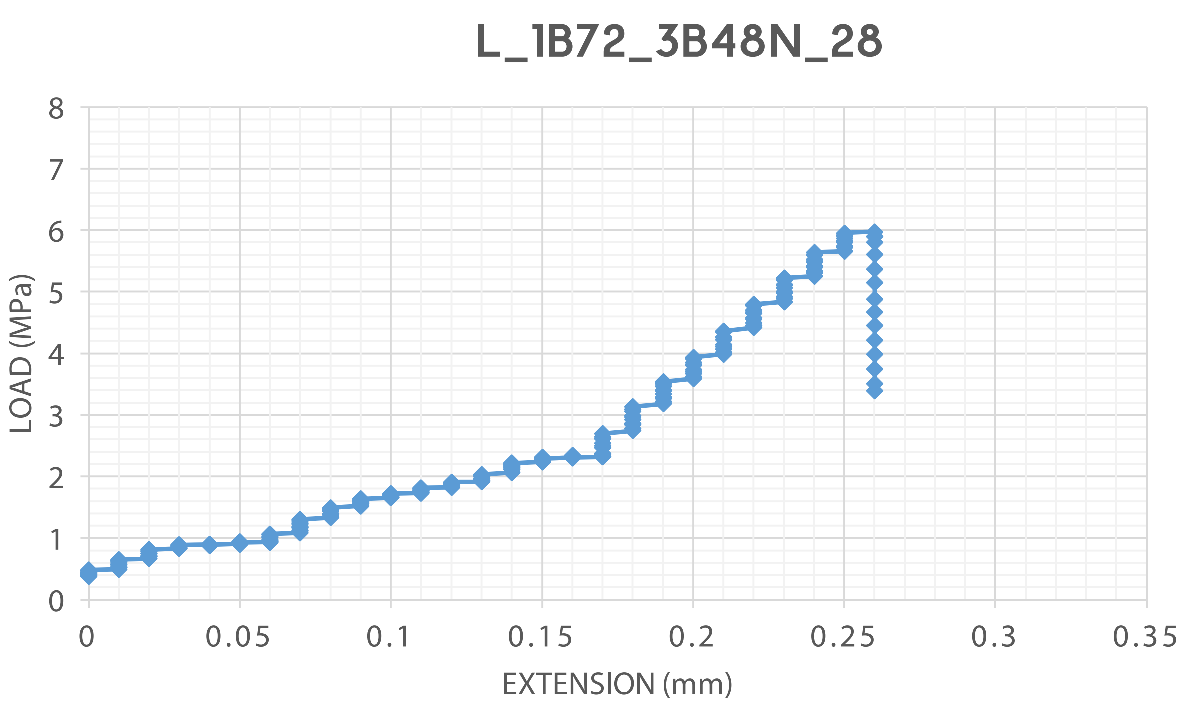
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_27
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35


L_1B72_3B48N_28
LOAD (MPa)
EXTENSION (mm)
0
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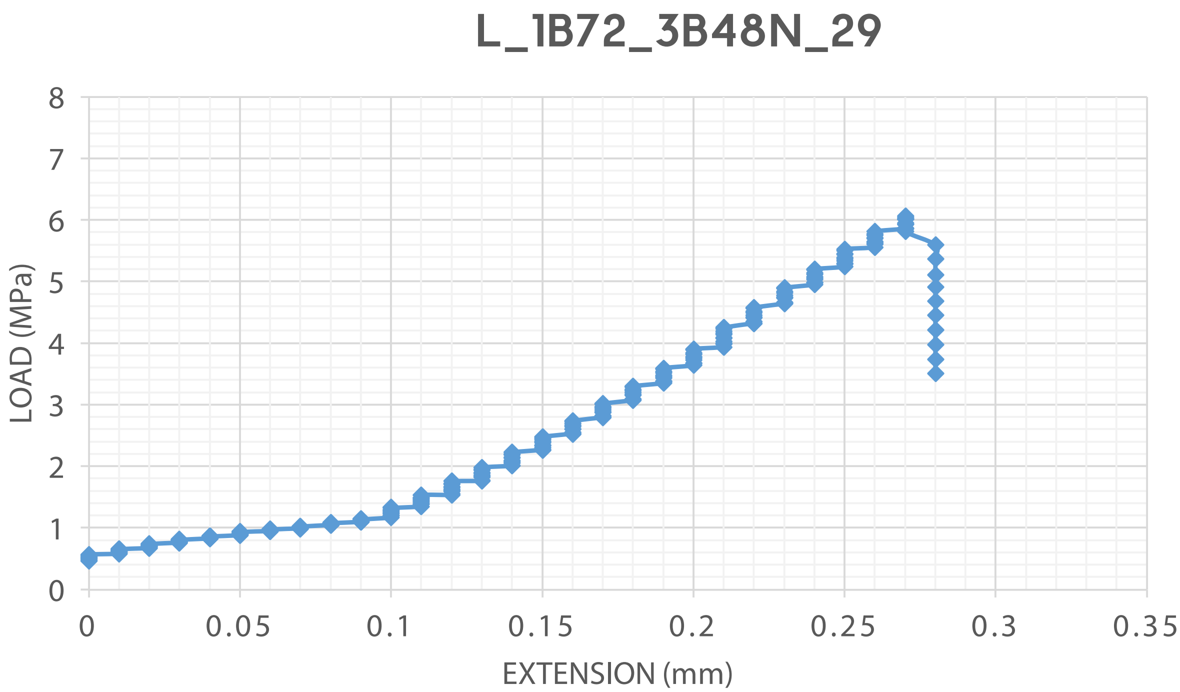
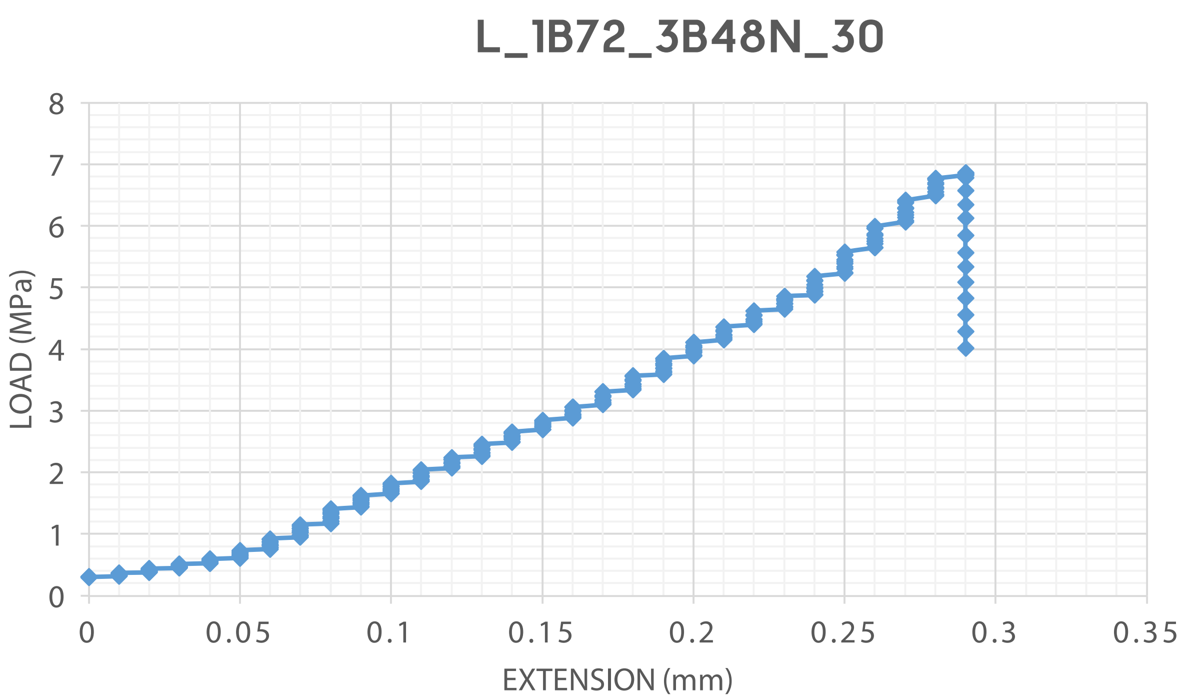
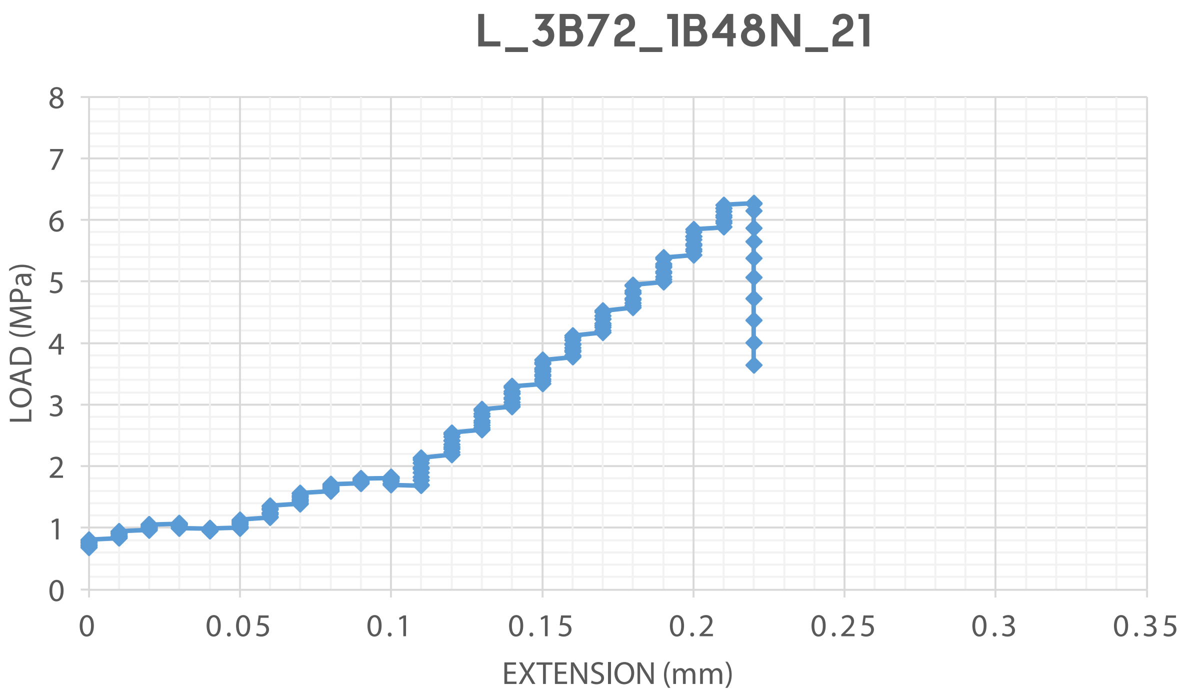
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35



L_1B72_3B48N_29
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35



L_1B72_3B48N_30
LOAD (MPa)
EXTENSION (mm)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35



L_3B72_1B48N_21
LOAD (MPa)
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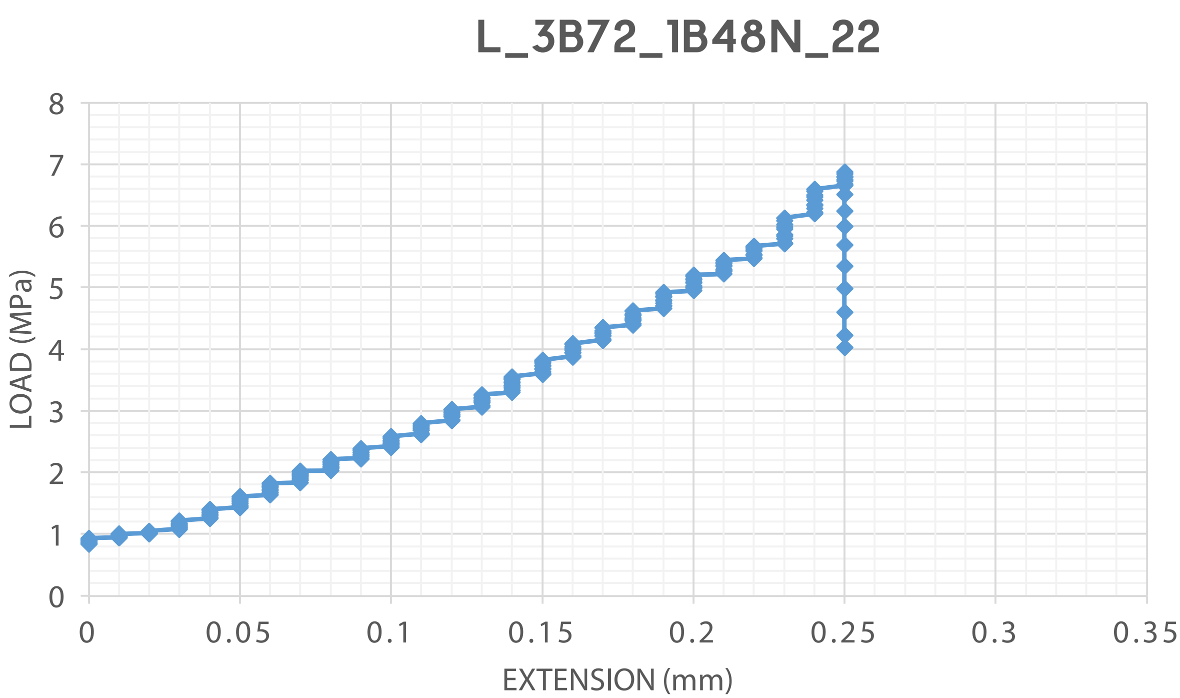
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)



L_3B72_1B48N_22
LOAD (MPa)
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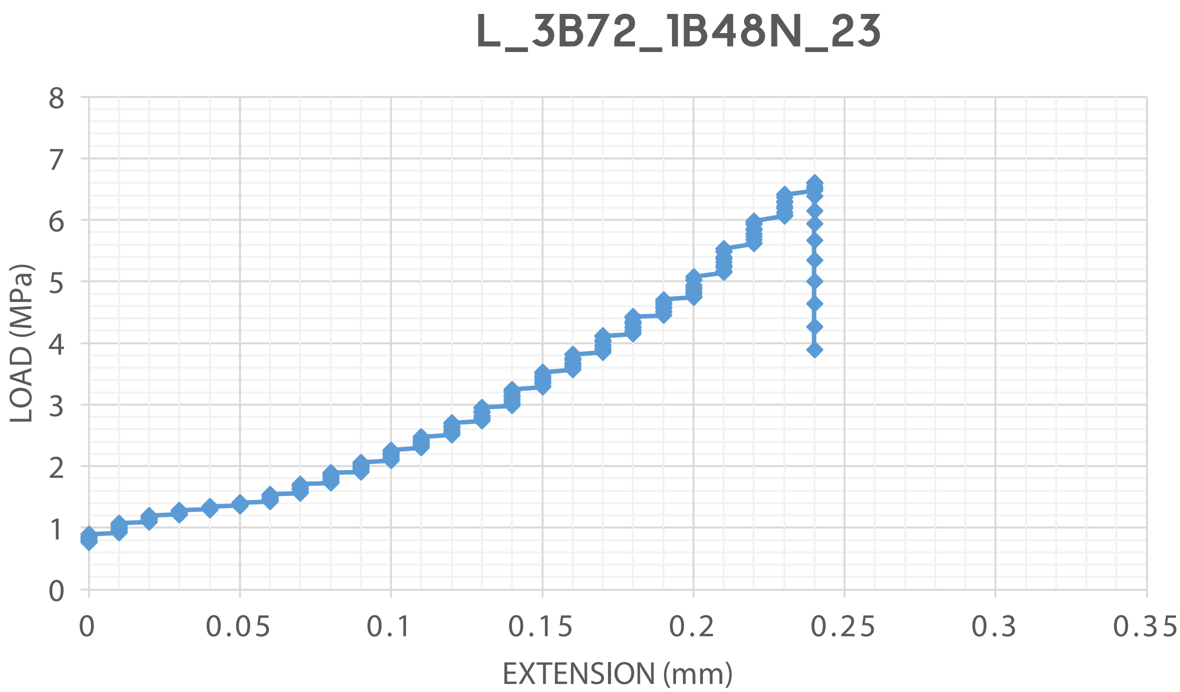
7
6
5
4
3
2
1
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_3B72_1B48N_23
LOAD (MPa)
0
1
2
3
4
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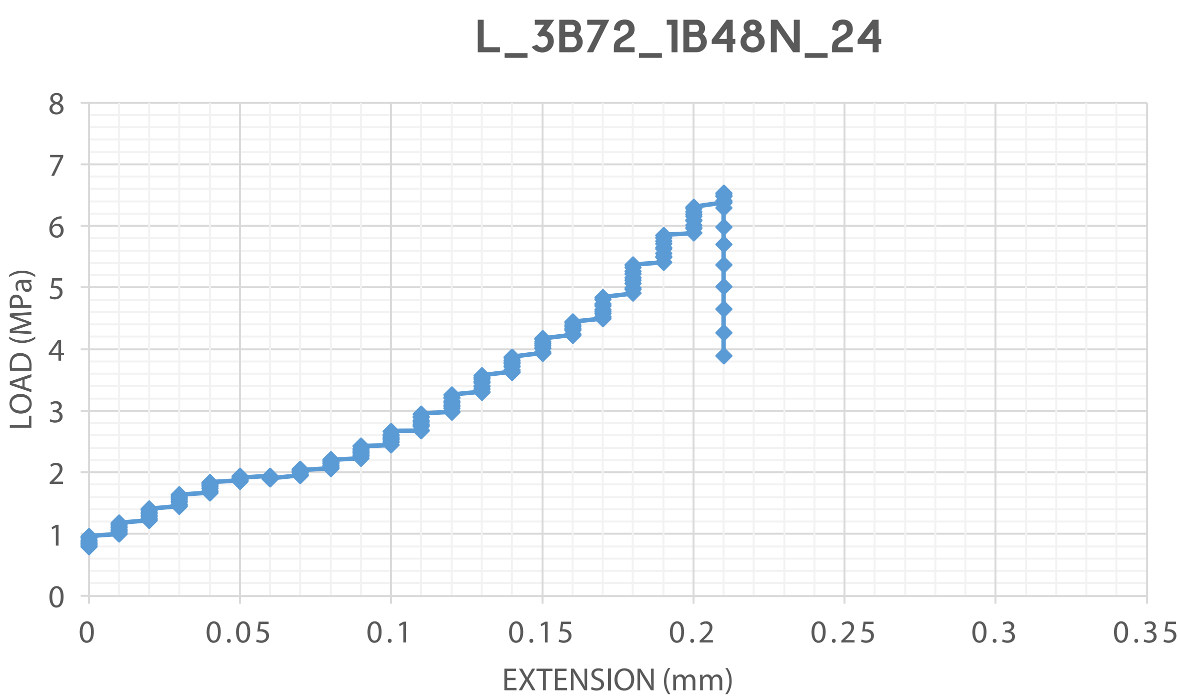
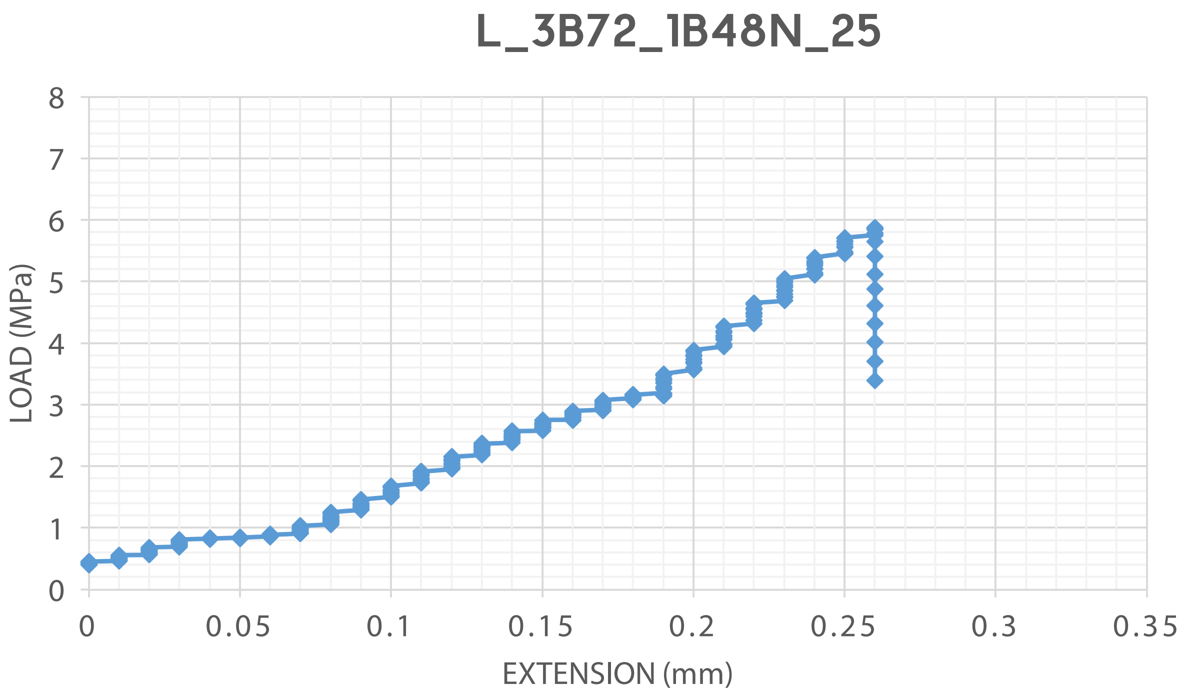
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_3B72_1B48N_24
LOAD (MPa)
0
1
2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
EXTENSION (mm)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0.25
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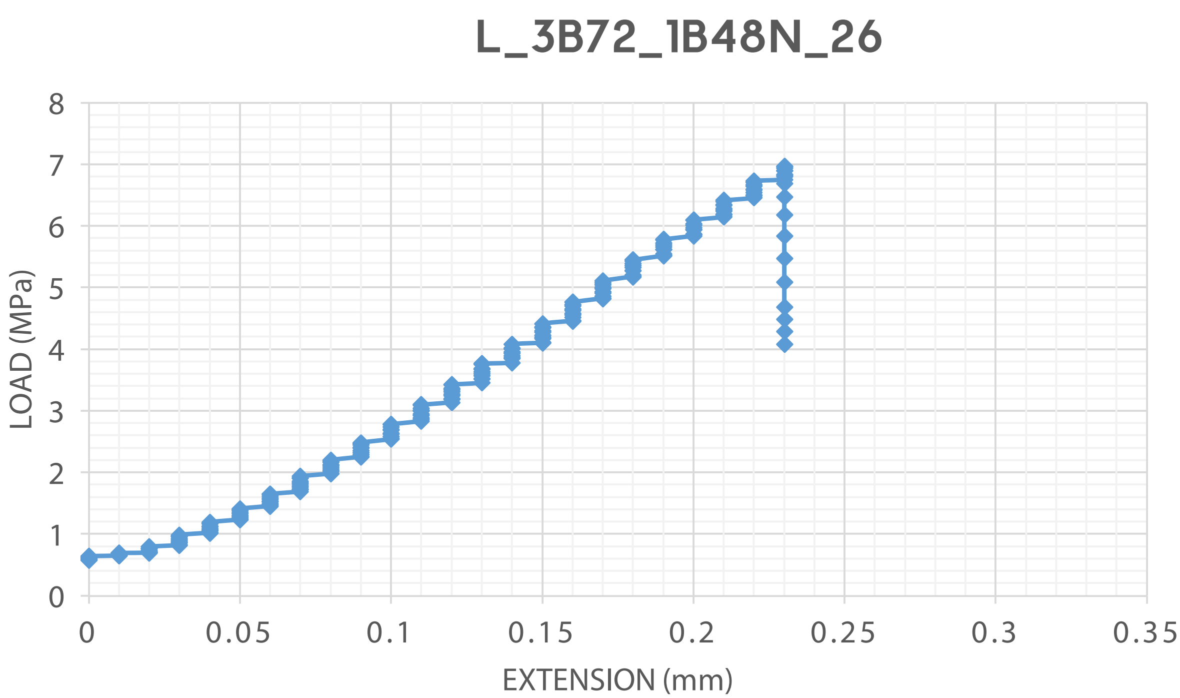
EXTENSION (mm)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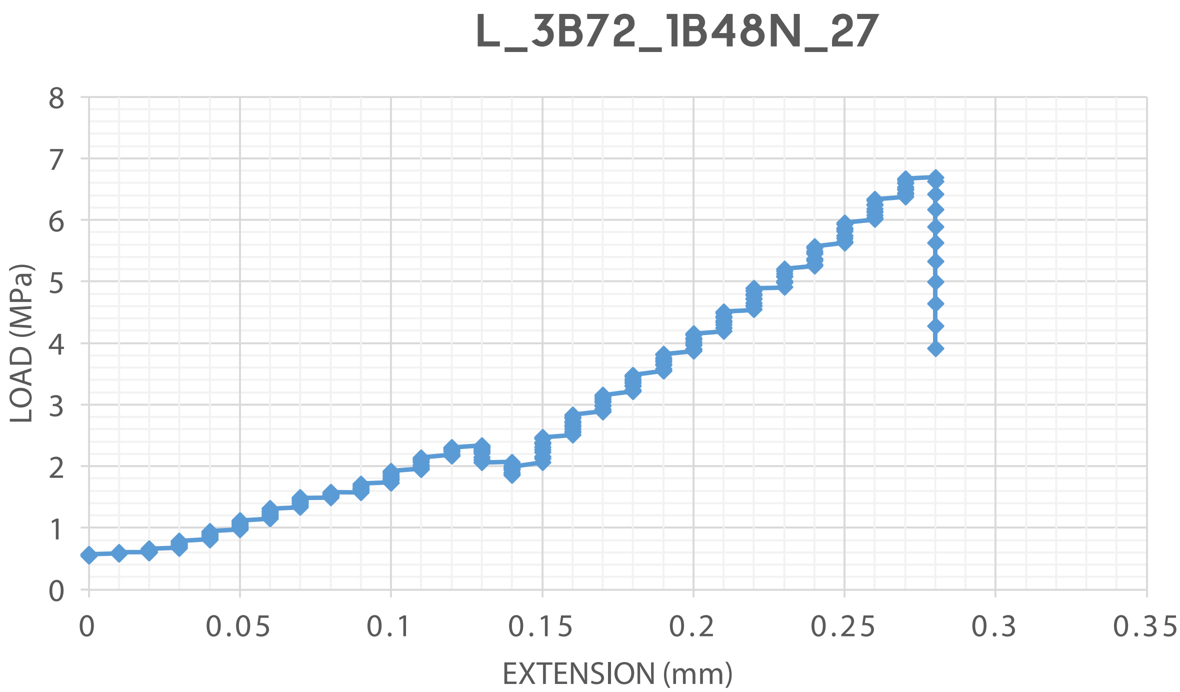
EXTENSION (mm)


L_3B72_1B48N_27
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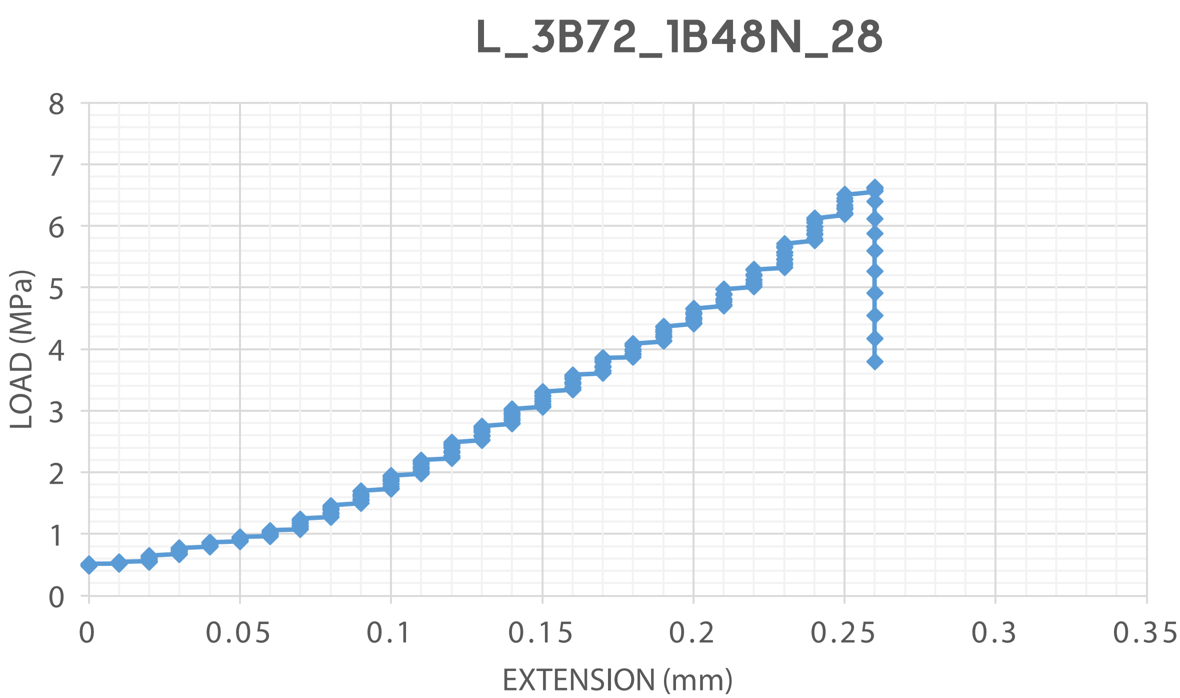
0.35
EXTENSION (mm)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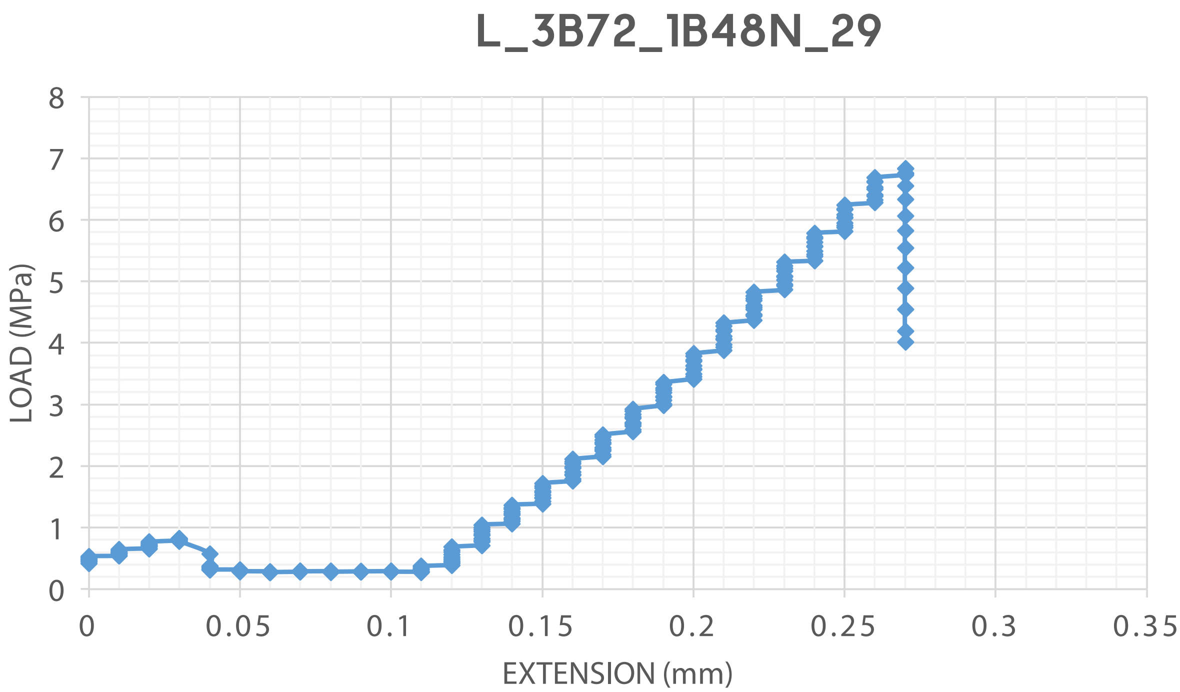
0.35
EXTENSION (mm)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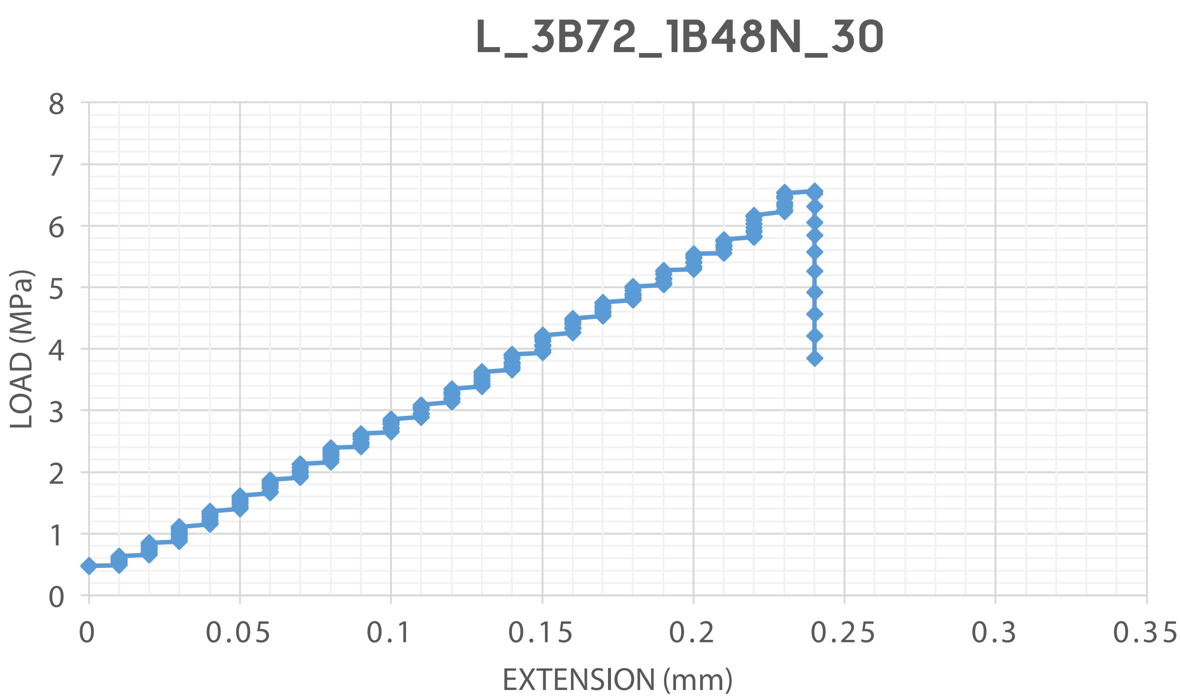
EXTENSION (mm)


L_3B72_1B48N_30
LOAD (MPa)
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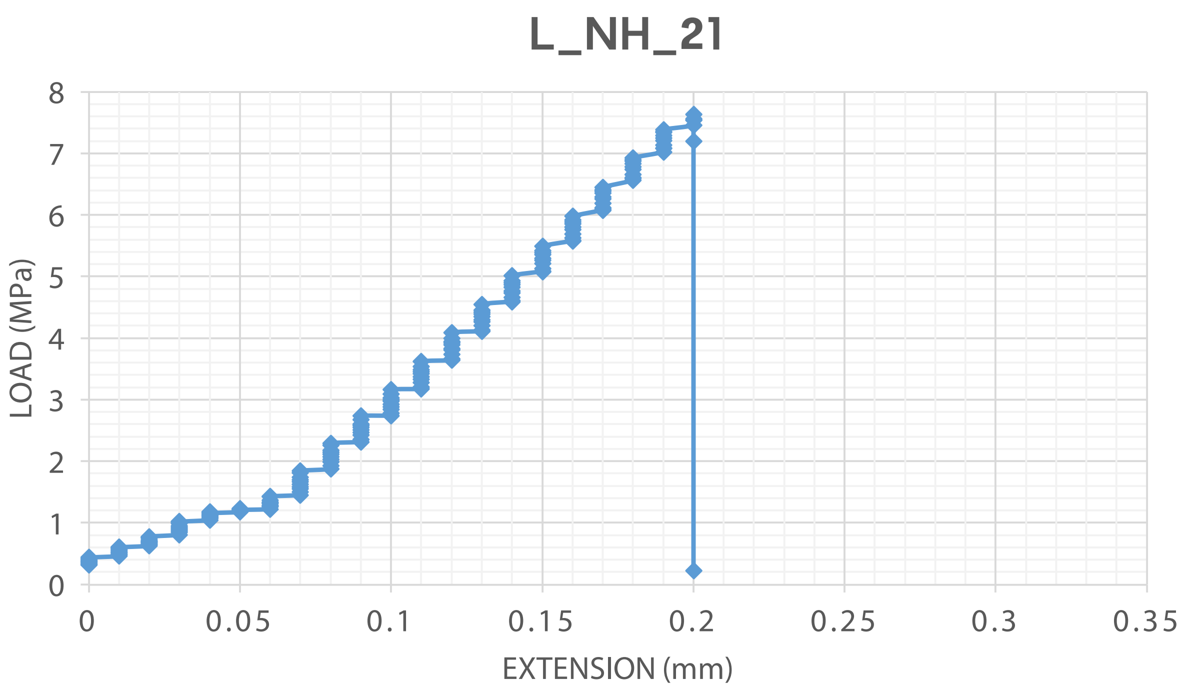
EXTENSION (mm)



L_NH_21
LOAD (MPa)
EXTENSION (mm)
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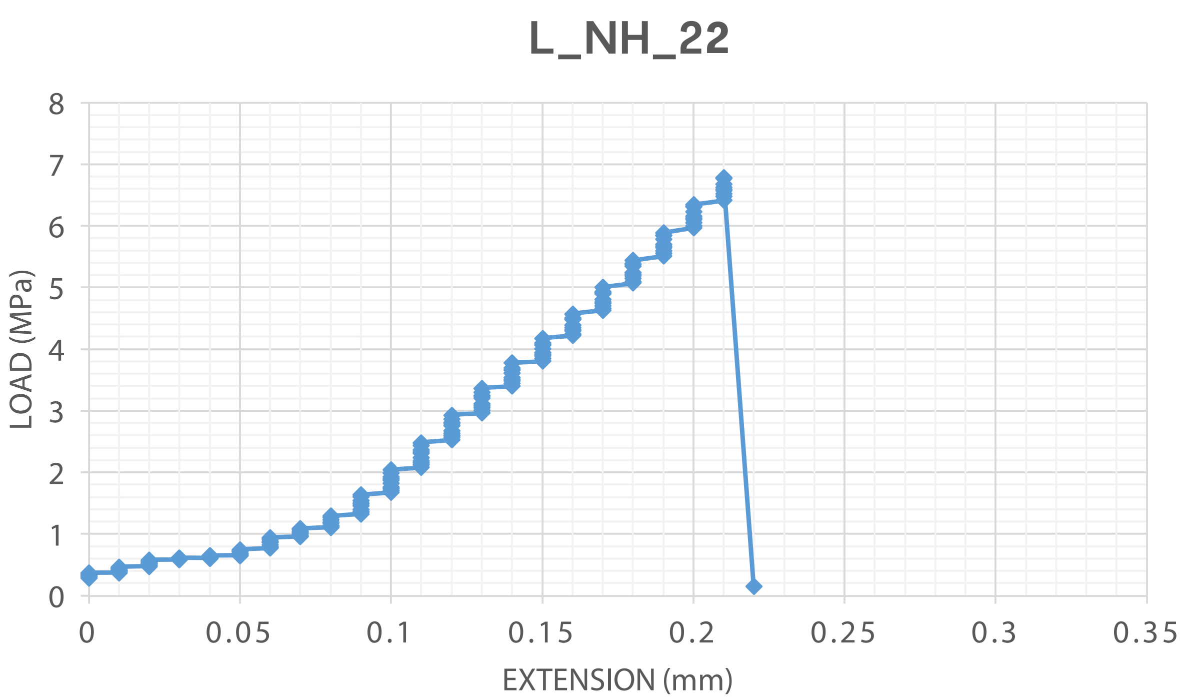
0.35



L_NH_22
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EXTENSION (mm)
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0.05
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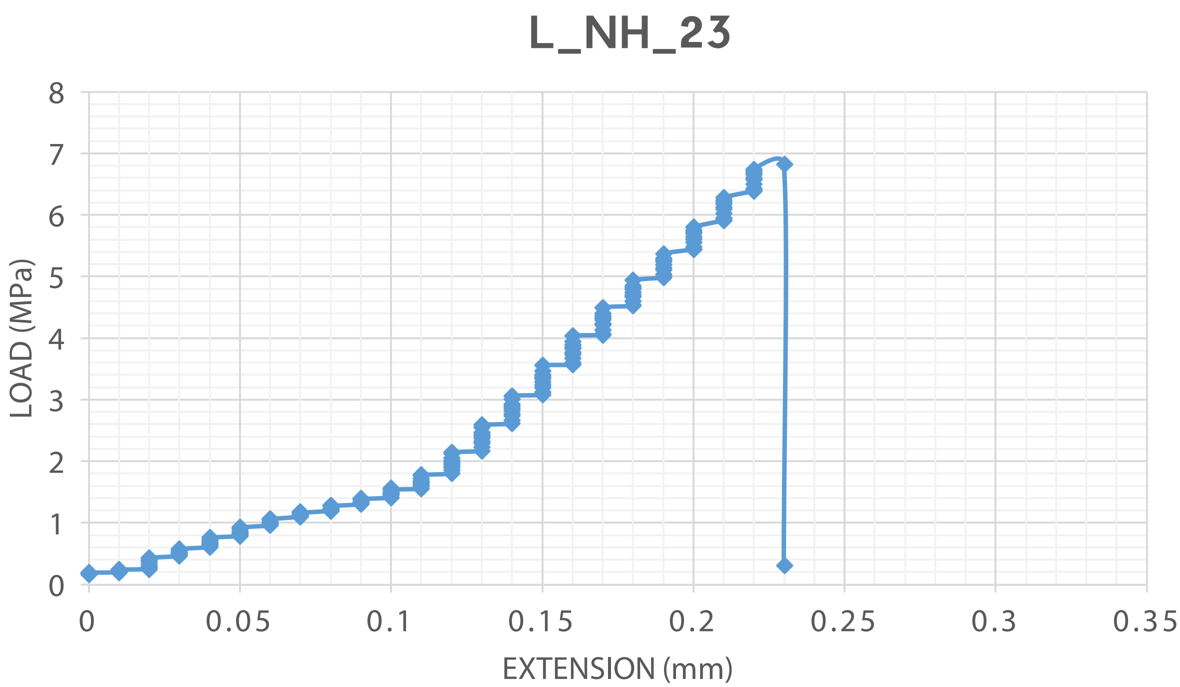
0.35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EXTENSION (mm)
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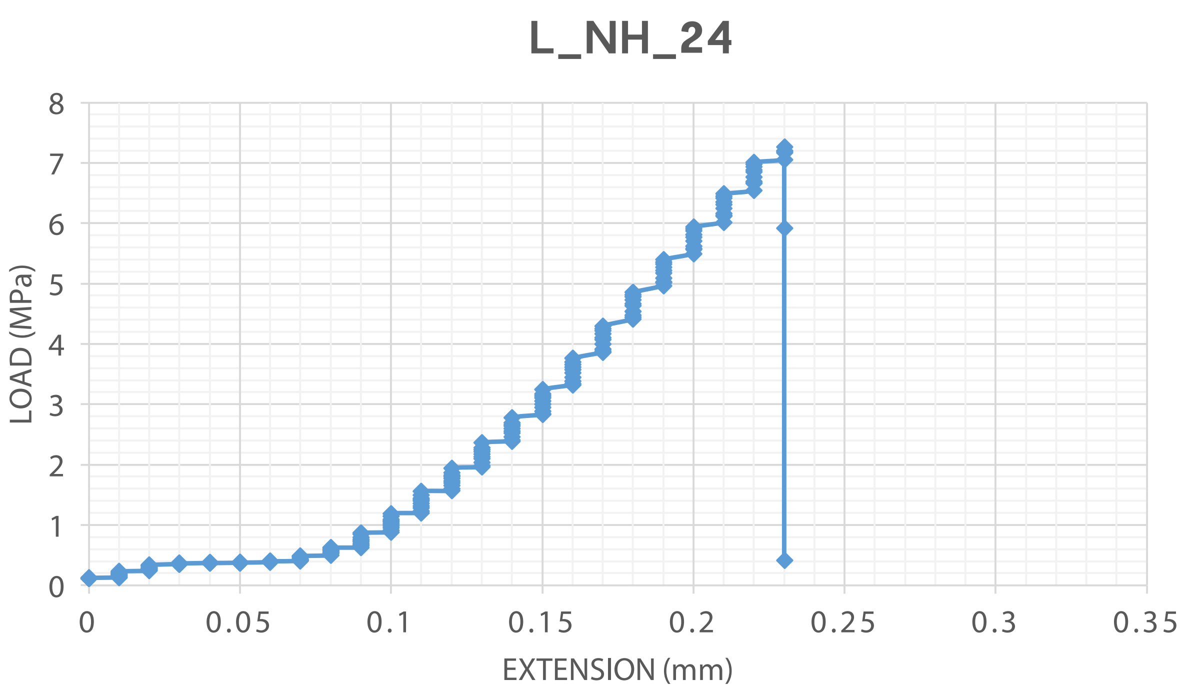
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EXTENSION (mm)
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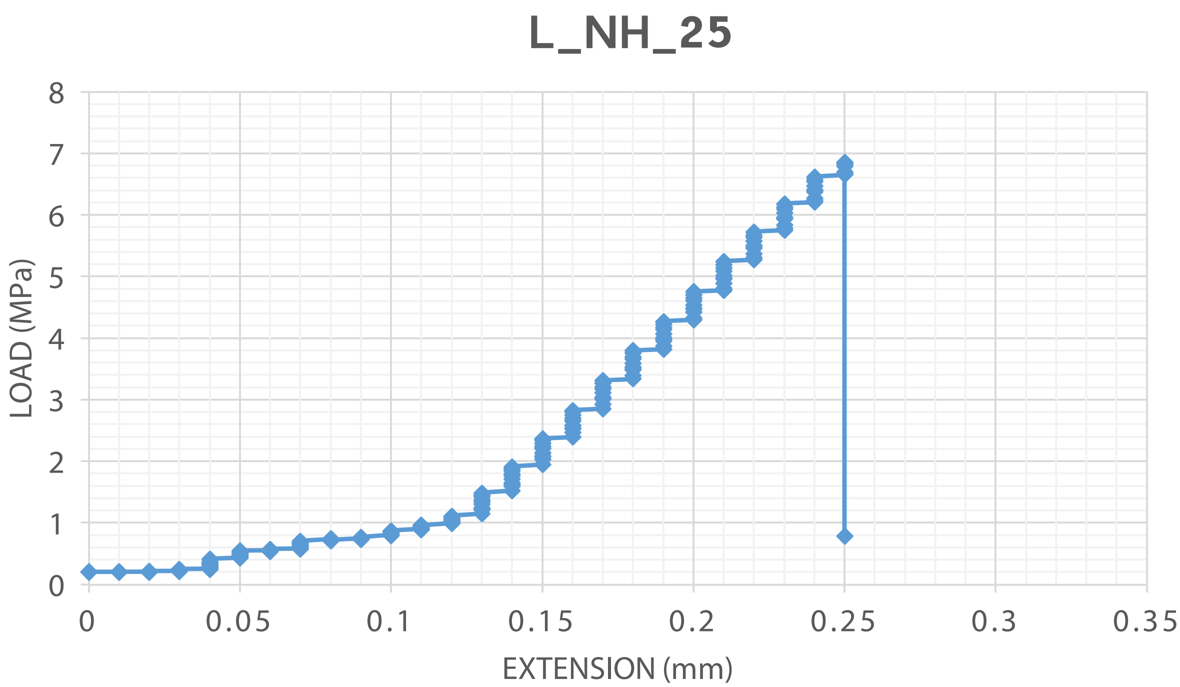
L_NH_25
LOAD (MPa)
EXTENSION (mm)
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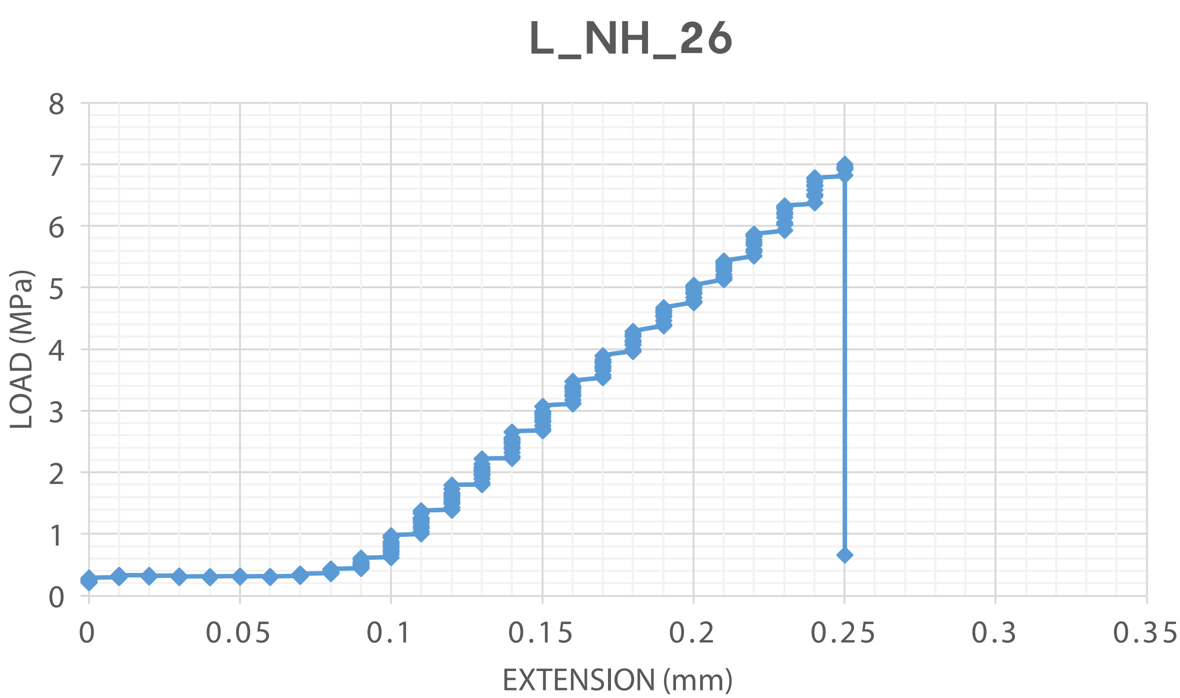
L_NH_26
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EXTENSION (mm)
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0.05
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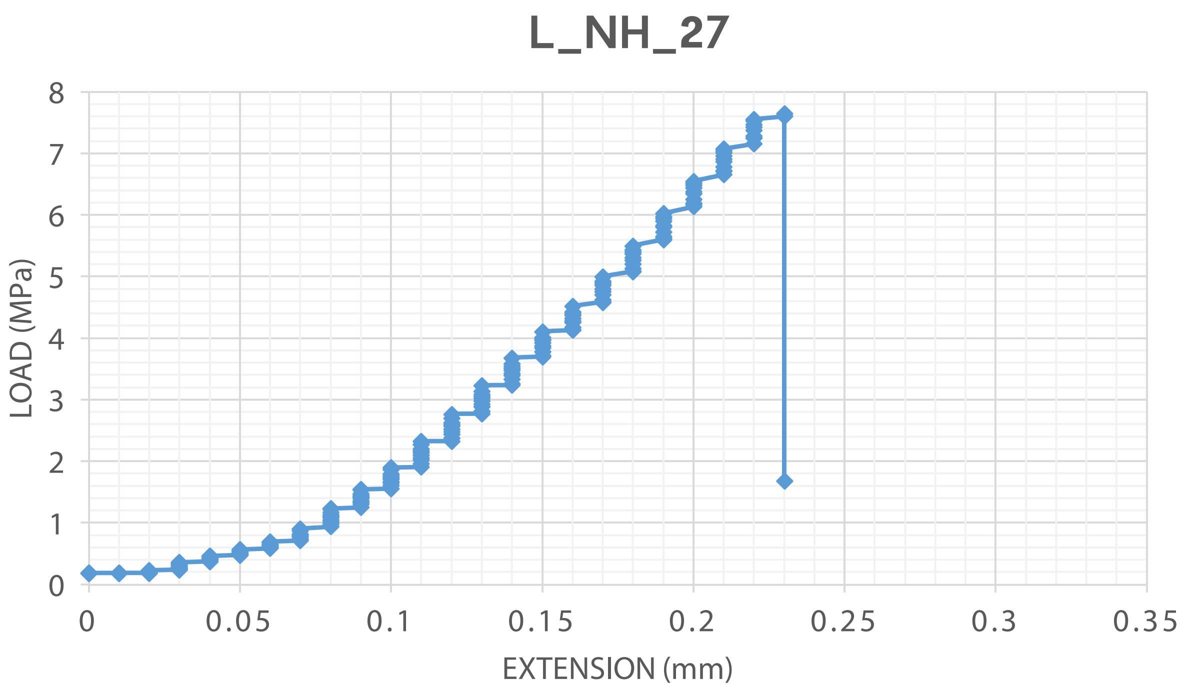
0.2
0.25
0.3
0.35


L_NH_27
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3
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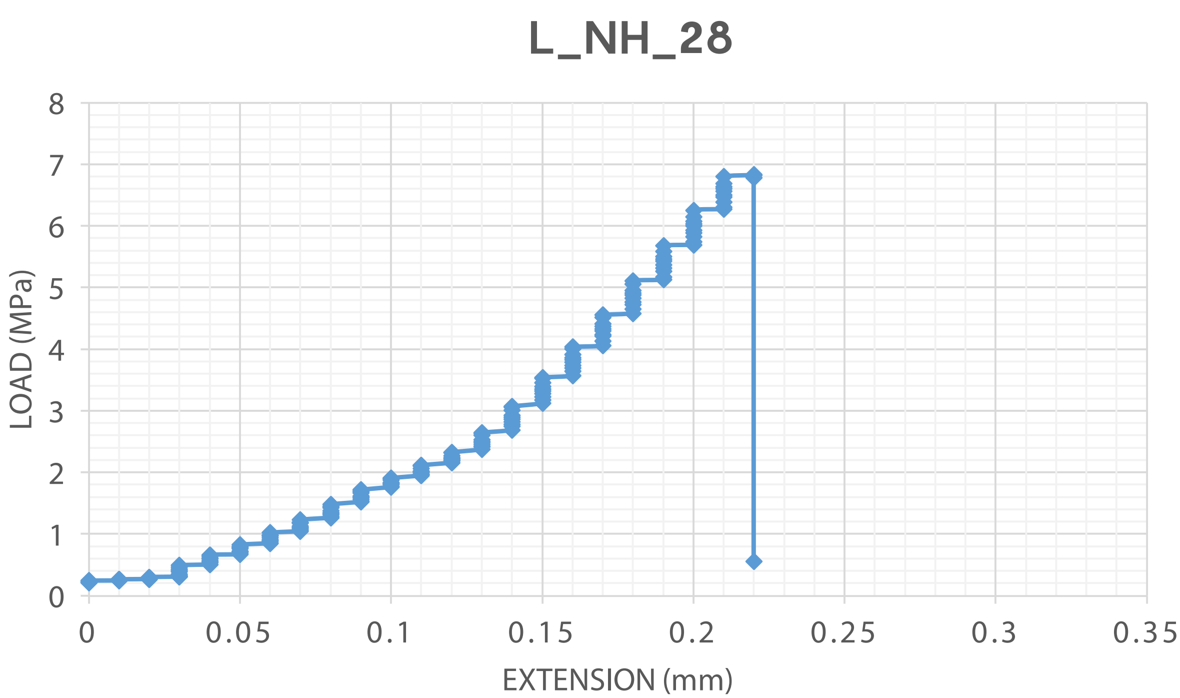
0.2
0.25
0.3
0.35
EXTENSION (mm)


L_NH_28
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0.1
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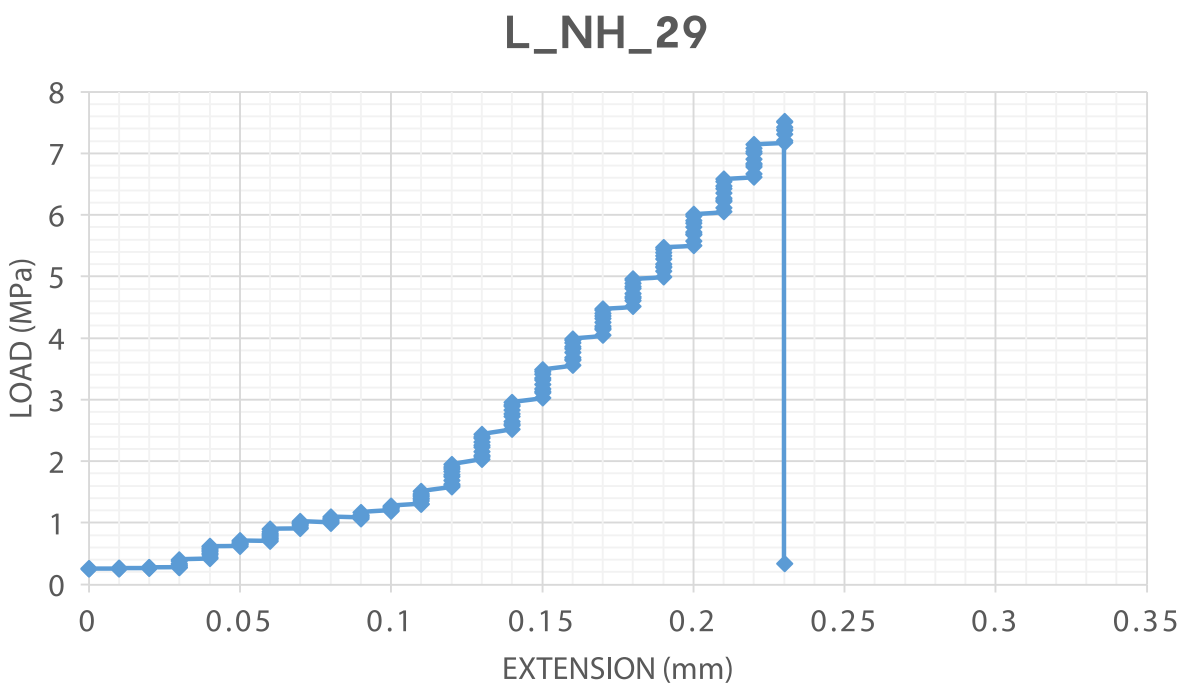
0.25
0.3
0.35
EXTENSION (mm)


L_NH_29
LOAD (MPa)
EXTENSION (mm)
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3
4
5
6
7
8
0
0.05
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0.15
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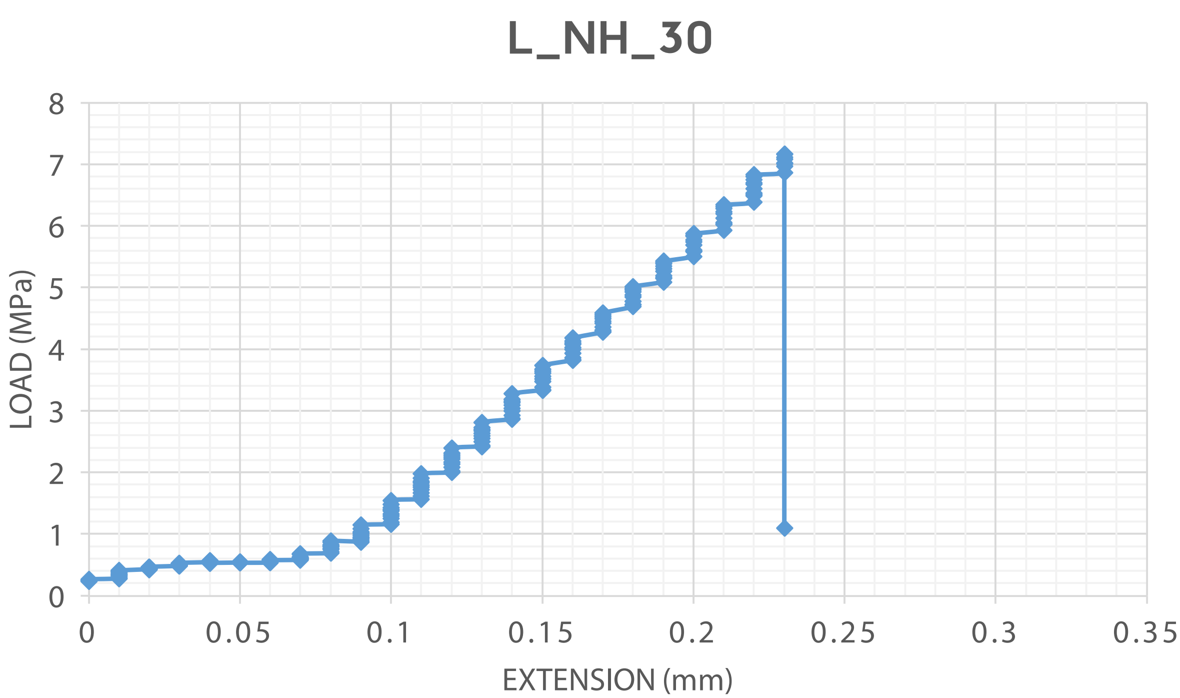
0.35


L_NH_30
LOAD (MPa)
EXTENSION (mm)
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2
3
4
5
6
7
8
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35

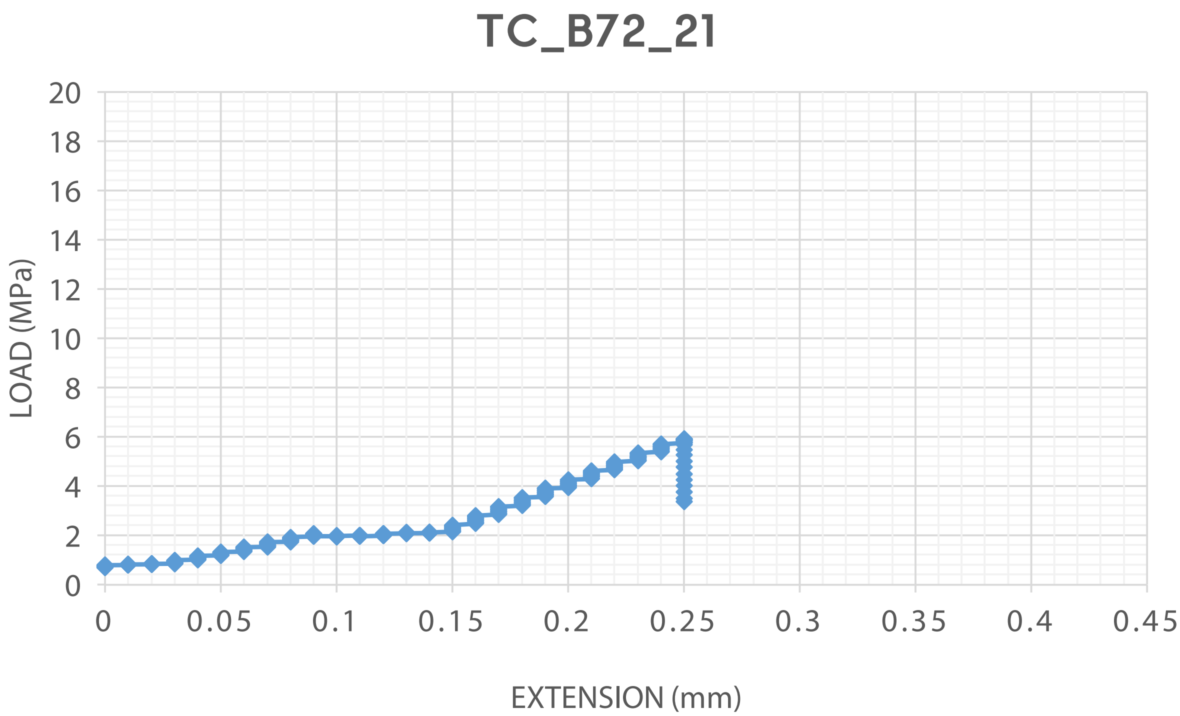
TERRA COTTA – GROUP C

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_B72_21	4/7/2017	4/24/2017	5/1/2017	369.67	No visible failure	6.06			1035.74
TC_B72_22	4/7/2017	4/24/2017	5/1/2017	364.61	Broke next to joint	5.98			1106.01
TC_B72_23	4/7/2017	4/24/2017	5/1/2017	376.63	Broke far from joint	6.18			1031.08
TC_B72_24	4/7/2017	4/24/2017	5/1/2017	509.82	Broke far from joint	8.36			1107.27
TC_B72_25	4/7/2017	4/24/2017	5/1/2017	593.45	Broke far from joint	9.73			1545.36
TC_B72_26	4/7/2017	4/24/2017	5/1/2017	573.61	No visible failure	9.41			1383.51
TC_B72_27	4/7/2017	4/24/2017	5/1/2017	779.86	No visible failure	12.79			1929.49
TC_B72_28	4/7/2017	4/24/2017	5/1/2017	694.66	Broke far from joint	11.39			1521.31
TC_B72_29	4/7/2017	4/24/2017	5/1/2017	467.27	No visible failure	7.66			1206.53
TC_B72_30	4/7/2017	4/24/2017	5/1/2017	826.79	Broke far from joint	13.56			1723.01
							9.11	2.78	
TC_B48N_21	4/7/2017	4/24/2017	5/1/2017	423.59	No visible failure	6.95			963.26
TC_B48N_22	4/7/2017	4/24/2017	5/1/2017	515.25	Broke next to joint	8.45			1187.67
TC_B48N_23	4/7/2017	4/24/2017	5/1/2017	437.45	No visible failure	7.17			848.23
TC_B48N_24	4/7/2017	4/24/2017	5/1/2017	610.2	Broke far from joint	10.01			1259.93
TC_B48N_25	4/7/2017	4/24/2017	5/1/2017	377.51	Broke next to joint	6.19			982.24
TC_B48N_26	4/7/2017	4/24/2017	5/1/2017	635.66	Broke next to joint	10.42			1409.01
TC_B48N_27	4/7/2017	4/24/2017	5/1/2017	728.22	Broke far from joint	11.94			1455.66
TC_B48N_28	4/7/2017	4/24/2017	5/1/2017	892.65	Broke AT joint	14.64			1891.59
TC_B48N_29	4/7/2017	4/24/2017	5/1/2017	614.96	Broke next to joint	10.09			1215.77
TC_B48N_30	4/7/2017	4/24/2017	5/1/2017	454.71	Broke far from joint	7.46			1726.32
							9.33	2.62	

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_B44_21	4/7/2017	4/24/2017	5/1/2017	920.02	Broke far from joint	15.09			1685.18
TC_B44_22	4/7/2017	4/24/2017	5/1/2017	385.76	Broke at flaw	6.33			832.96
TC_B44_23	4/7/2017	4/24/2017	5/1/2017	382.74	No visible failure	6.28			1016.03
TC_B44_24	4/7/2017	4/24/2017	5/1/2017	297.29	No visible failure	4.88			1478.11
TC_B44_25	4/7/2017	4/24/2017	5/1/2017	873.39	Broke next to joint	14.32			1729.35
TC_B44_26	4/7/2017	4/24/2017	5/1/2017	772.85	Broke far from joint	12.67			1676.48
TC_B44_27	4/7/2017	4/24/2017	5/1/2017	612.96	No visible failure	10.05			1408.16
TC_B44_28	4/7/2017	4/24/2017	5/1/2017	615.1	Broke far from joint	10.09			1343.40
TC_B44_29	4/7/2017	4/24/2017	5/1/2017	654.92	Broke far from joint	10.74			1248.78
TC_B44_30	4/7/2017	4/24/2017	5/1/2017	841.57	Broke next to joint	13.80			1980.90
							10.42	3.63	
TC_A11_21	4/7/2017	4/24/2017	5/1/2017	785.01	Broke next to joint	12.87			1829.60
TC_A11_22	4/7/2017	4/24/2017	5/1/2017	596.79	Broke far from joint	9.79			935.06
TC_A11_23	4/7/2017	4/24/2017	5/1/2017	435.62	Failed at flaw	7.14			1153.54
TC_A11_24	4/7/2017	4/24/2017	5/1/2017	520.73	No visible failure	8.54			1005.69
TC_A11_25	4/7/2017	4/24/2017	5/1/2017	480.18	No visible failure	7.87			1326.58
TC_A11_26	4/7/2017	4/24/2017	5/1/2017	464.23	Broke next to joint	7.61			1135.39
TC_A11_27	4/7/2017	4/24/2017	5/1/2017	875.45	Broke far from joint	14.36			2114.74
TC_A11_28	4/7/2017	4/24/2017	5/1/2017	330.64	No visible failure	5.42			568.12
TC_A11_29	4/7/2017	4/24/2017	5/1/2017	644.04	Broke far from joint	10.56			1266.92
TC_A11_30	4/7/2017	4/24/2017	5/1/2017	1004.08	Broke next to joint	16.47			1896.55
							10.06	3.51	
TC_1B72_3B48N_21	4/7/2017	4/24/2017	5/1/2017	480.68	No visible failure	7.88			949.80

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_1B72_3B48N_22	4/7/2017	4/24/2017	5/1/2017	949.64	Broke next to joint	15.57			1498.29
TC_1B72_3B48N_23	4/7/2017	4/24/2017	5/1/2017	570.84	No visible failure	9.36			1027.92
TC_1B72_3B48N_24	4/7/2017	4/24/2017	5/1/2017	315.68	Broke next to joint	5.18			963.90
TC_1B72_3B48N_25	4/7/2017	4/24/2017	5/1/2017	318.27	No visible failure	5.22			772.62
TC_1B72_3B48N_26	4/7/2017	4/24/2017	5/1/2017	839.17	Broke next to joint	13.76			1246.08
TC_1B72_3B48N_27	4/7/2017	4/24/2017	5/1/2017	543.45	Broke next to joint	8.91			1134.69
TC_1B72_3B48N_28	4/7/2017	4/24/2017	5/1/2017	656.62	Broke AT joint	10.77			1189.71
TC_1B72_3B48N_29	4/7/2017	4/24/2017	5/1/2017	532.07	Broke next to joint	8.73			1431.78
TC_1B72_3B48N_30	4/7/2017	4/24/2017	5/1/2017	476.81	Broke far from joint	7.82			1168.99
							9.32	3.33	
TC_3B72_1B48N_21	4/7/2017	4/24/2017	5/1/2017	532.71	Broke far from joint	8.74			1031.29
TC_3B72_1B48N_22	4/7/2017	4/24/2017	5/1/2017	574.17	No visible failure	9.42			1295.33
TC_3B72_1B48N_23	4/7/2017	4/24/2017	5/1/2017	588.54	Broke next to joint	9.65			1293.63
TC_3B72_1B48N_24	4/7/2017	4/24/2017	5/1/2017	515.18	No visible failure	8.45			1147.70
TC_3B72_1B48N_25	4/7/2017	4/24/2017	5/1/2017	413.31	No visible failure	6.78			1071.01
TC_3B72_1B48N_26	4/7/2017	4/24/2017	5/1/2017	657.06	Broke far from joint	10.78			1389.15
TC_3B72_1B48N_27	4/7/2017	4/24/2017	5/1/2017	654.36	Broke far from joint	10.73			1413.22
TC_3B72_1B48N_28	4/7/2017	4/24/2017	5/1/2017	520.51	Broke far from joint	8.54			1056.89
TC_3B72_1B48N_29	4/7/2017	4/24/2017	5/1/2017	561.3	Broke far from joint	9.21			1292.98
TC_3B72_1B48N_30	4/7/2017	4/24/2017	5/1/2017	402.81	Broke far from joint	6.61			1164.62
							8.89	1.41	
TC_CT_NH_21	N/A	N/A	4/5/2017	964.88		15.82			2023.59
TC_CT_NH_22	N/A	N/A	4/5/2017	710.79		11.66			1476.18

	Bonding Start Date	Bonding End Date	4-Point Bend Testing Date	Load Peak (N)	Notes	Stress (MPa)	Average	Standard Deviation	Modulus of Elasticity (MPa)
TC_CT_NH_23	N/A	N/A	4/5/2017	173.09		2.84			834.11
TC_CT_NH_24	N/A	N/A	4/5/2017	745.80		12.23			1808.21
TC_CT_NH_25	N/A	N/A	4/5/2017	370.69		6.08			801.79
TC_CT_NH_26	N/A	N/A	4/5/2017	544.17		8.92			1538.89
TC_CT_NH_27	N/A	N/A	4/5/2017	423.75		6.95			1385.29
TC_CT_NH_28	N/A	N/A	4/5/2017	466.01		7.64			1256.74
TC_CT_NH_29	N/A	N/A	4/5/2017	566.23		9.29			2024.85
TC_CT_NH_30	N/A	N/A	4/5/2017	626.06		10.27			1744.36
							9.17	3.62	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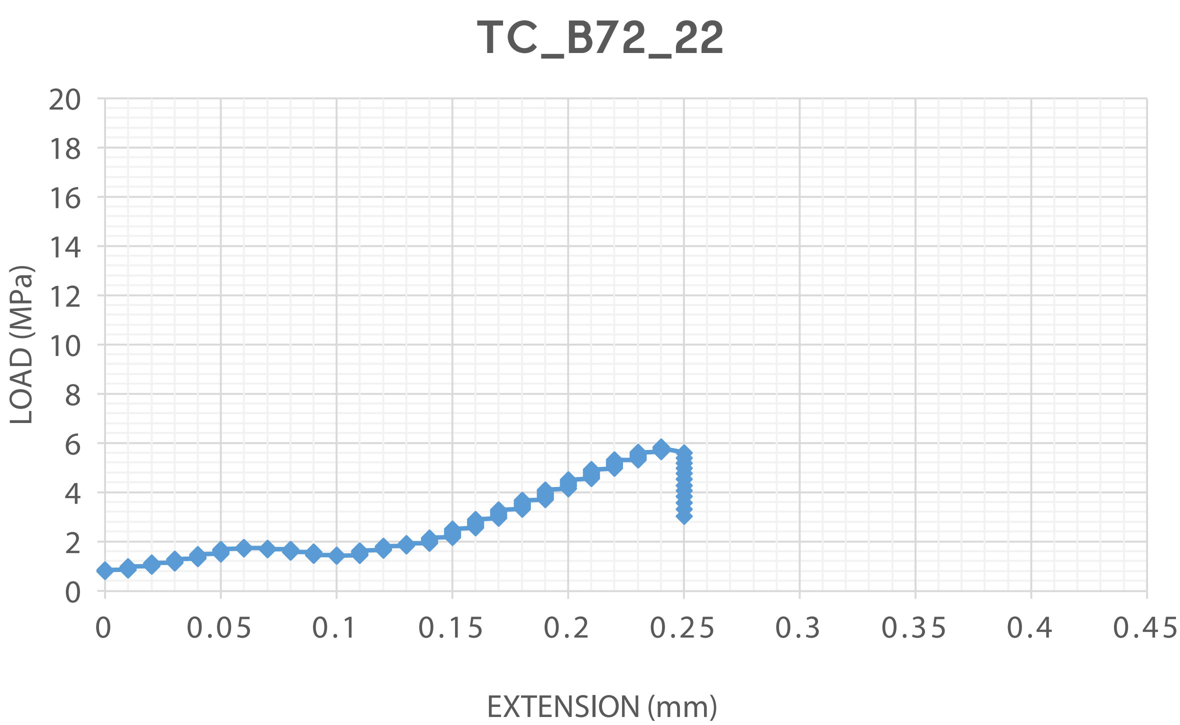
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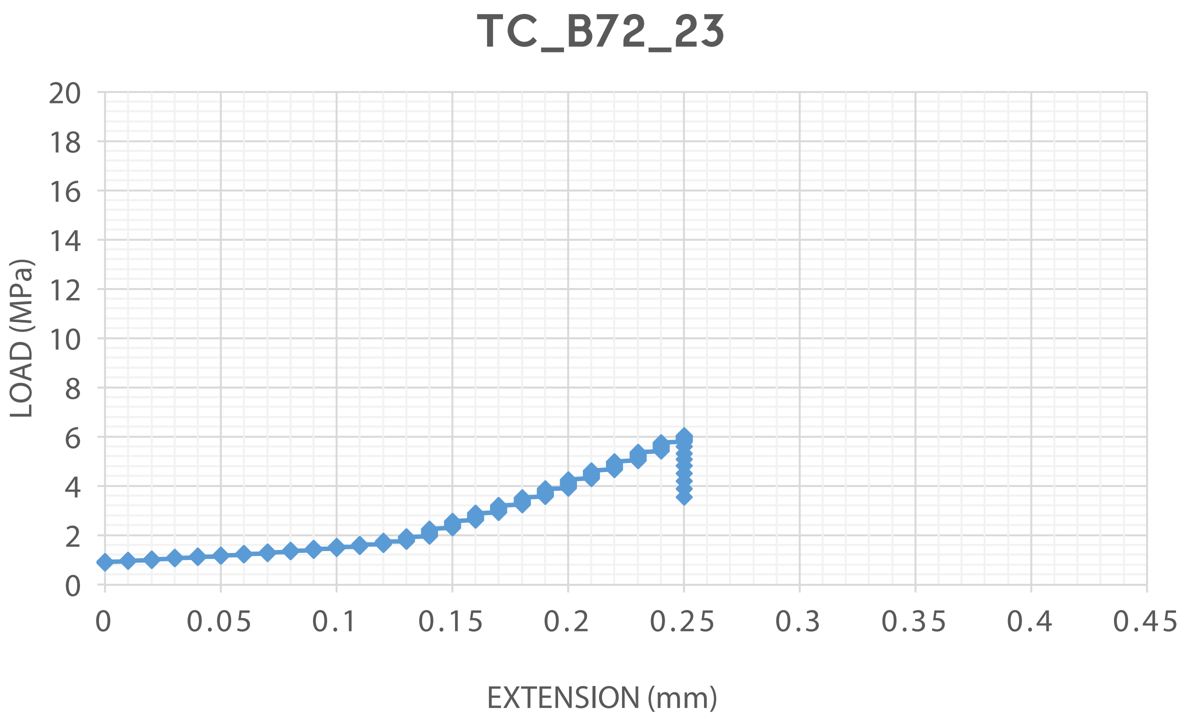
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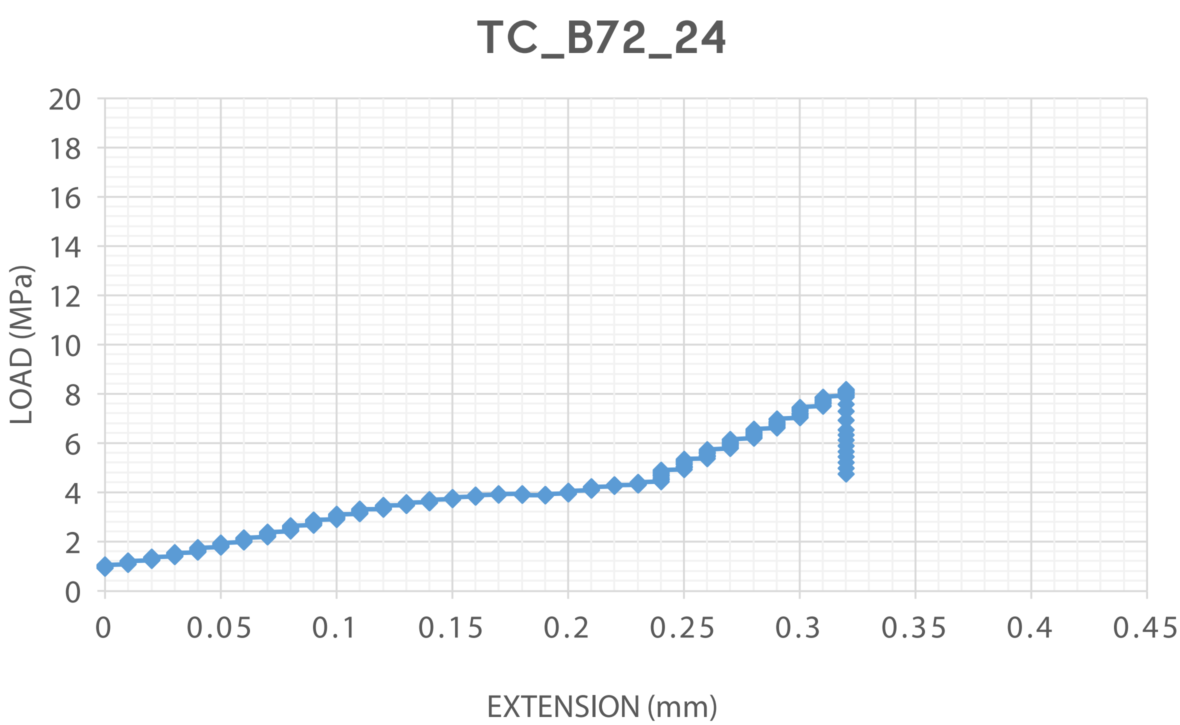
TC_B72_24
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EXTENSION (mm)
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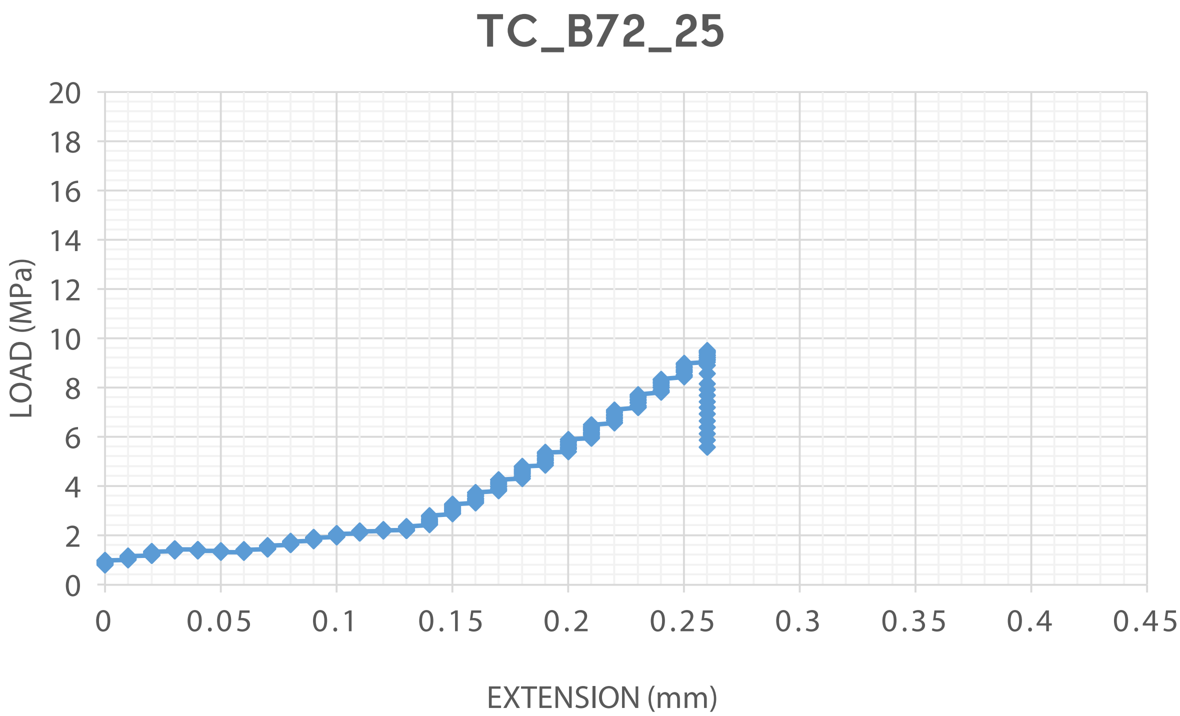
TC_B72_25
LOAD (MPa)
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10
8
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0.15
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0.25
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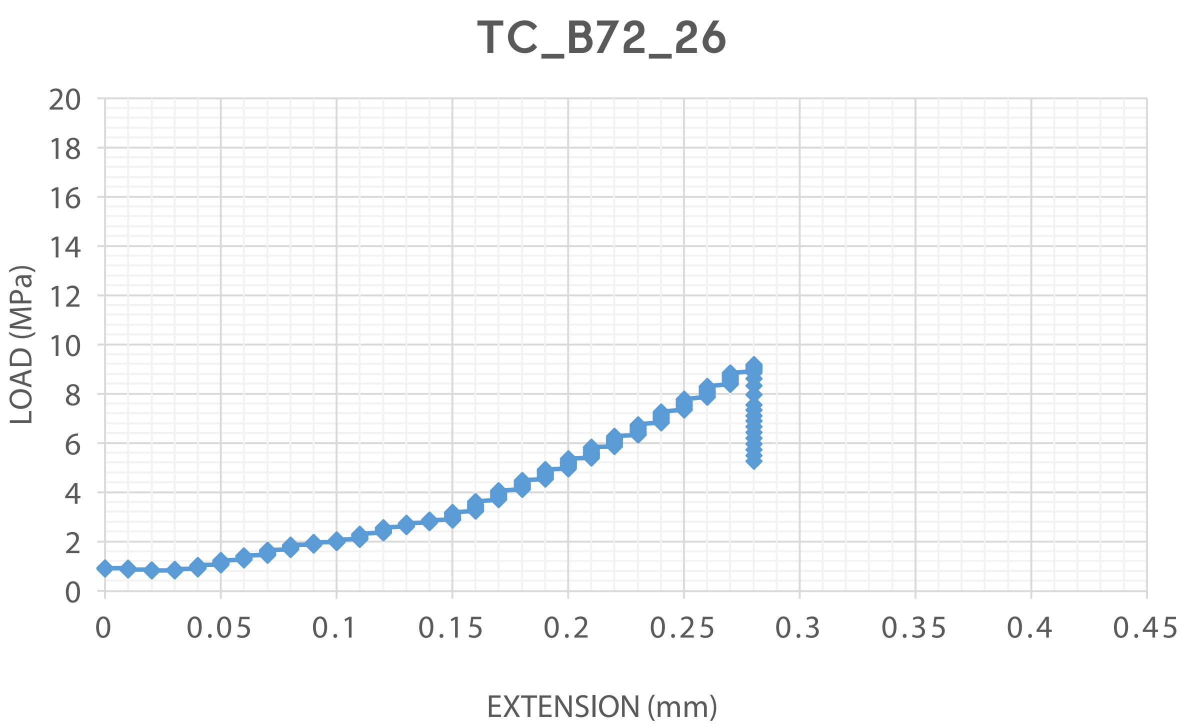
EXTENSION (mm)


TC_B72_26
LOAD (MPa)
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14
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10
8
6
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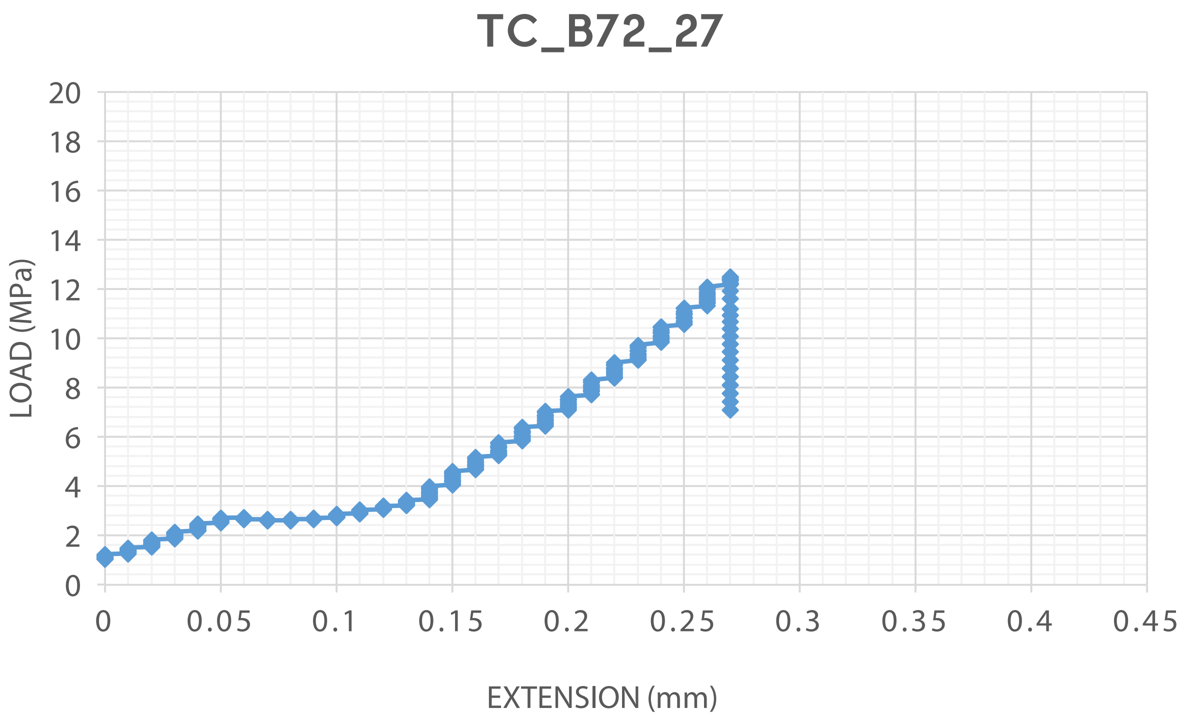
EXTENSION (mm)


TC_B72_27
LOAD (MPa)
20
18
16
14
12
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8
6
4
2
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0
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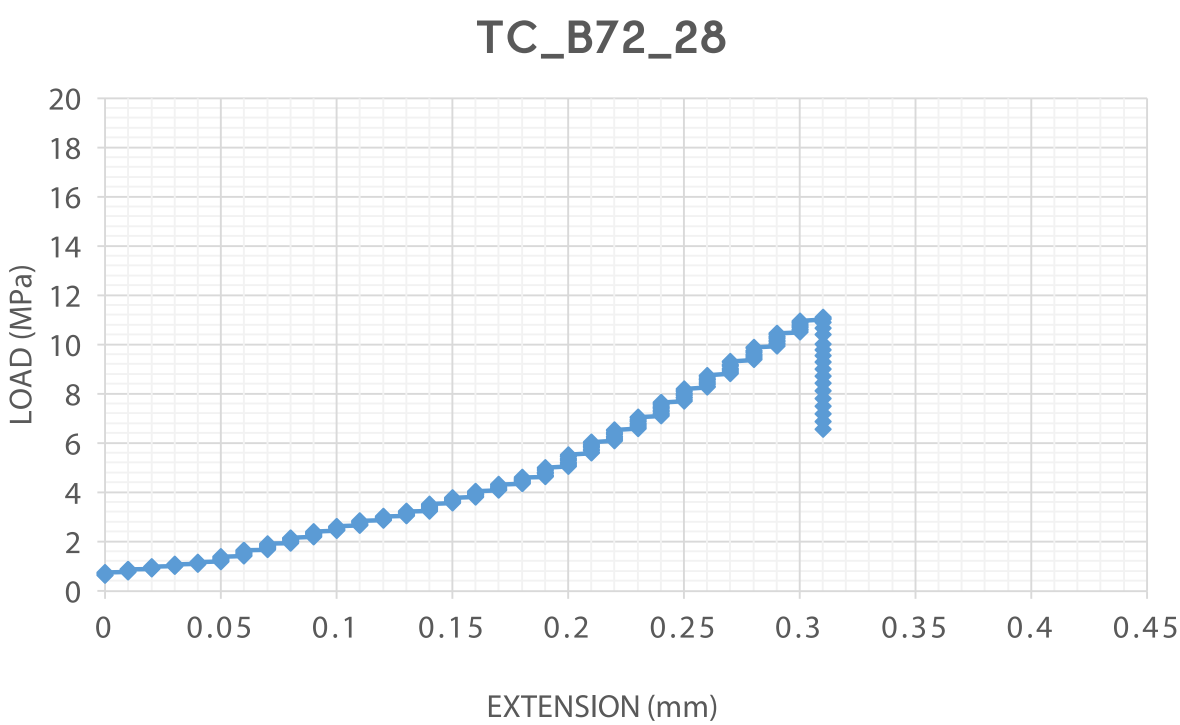
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)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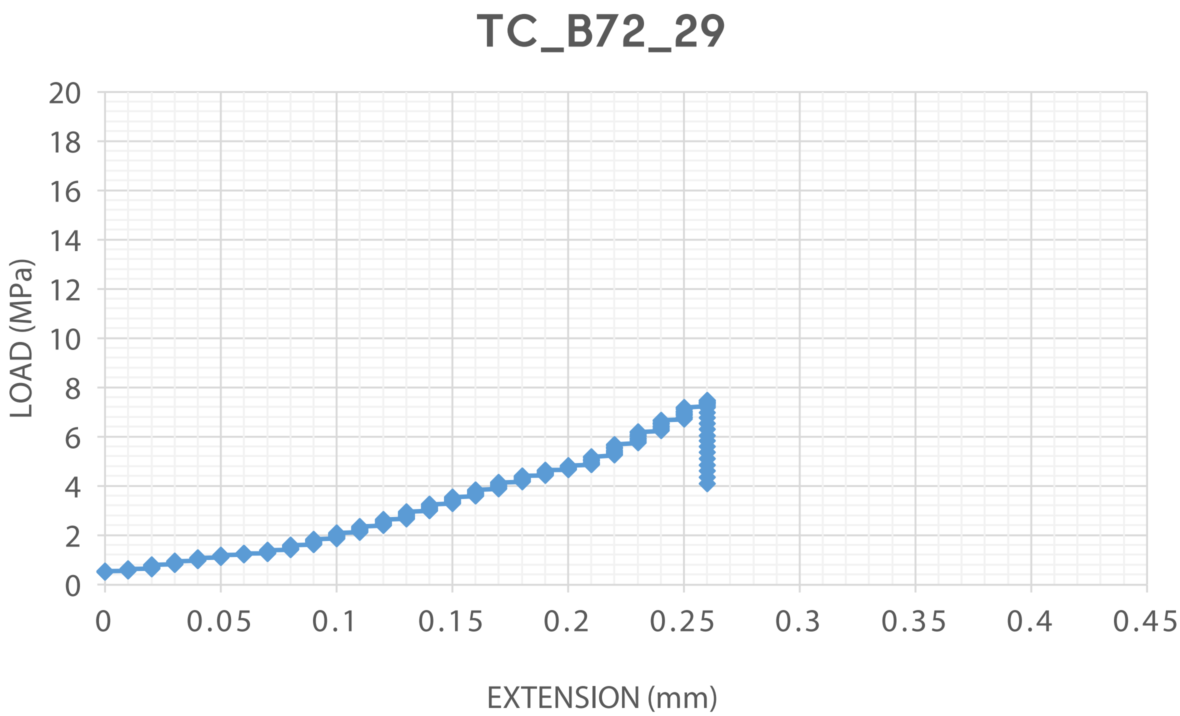
EXTENSION (mm)


TC_B72_29
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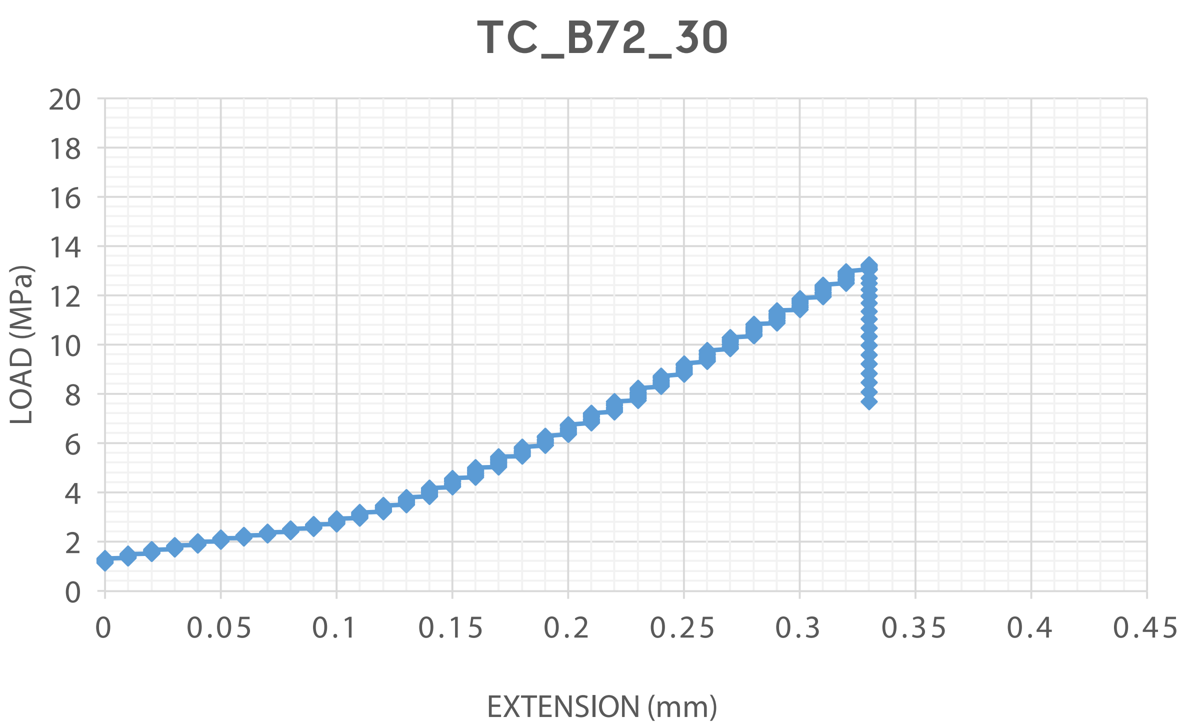
EXTENSION (mm)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0.35
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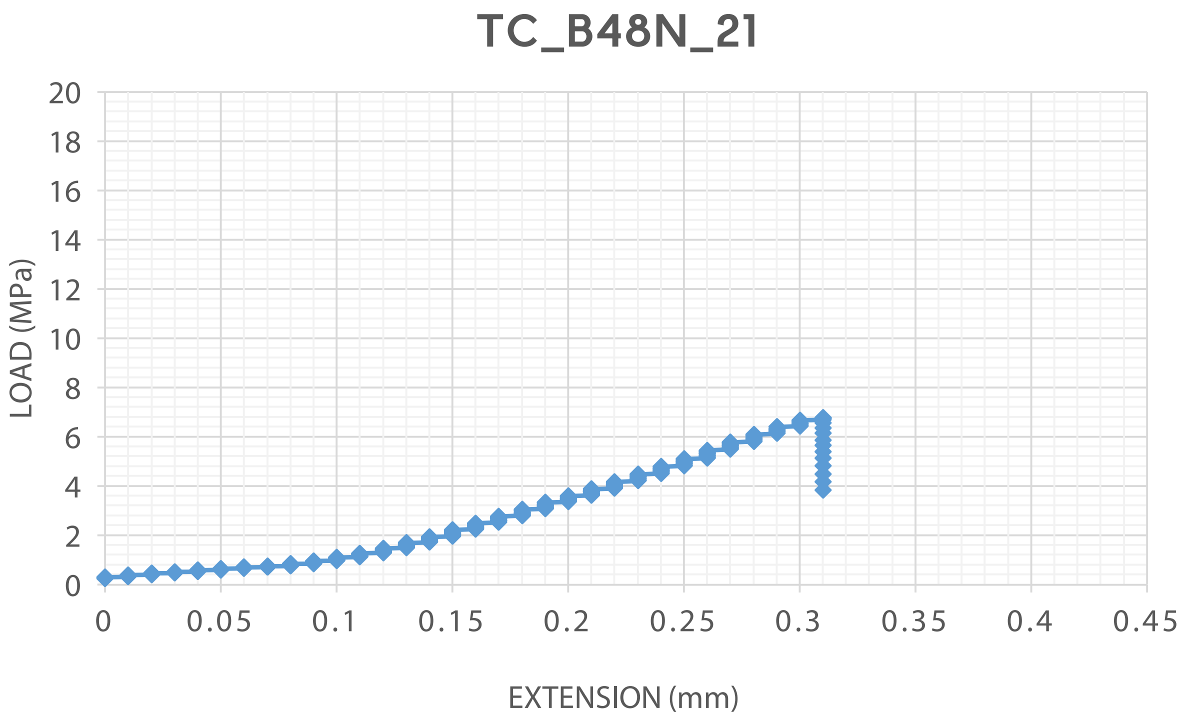
EXTENSION (mm)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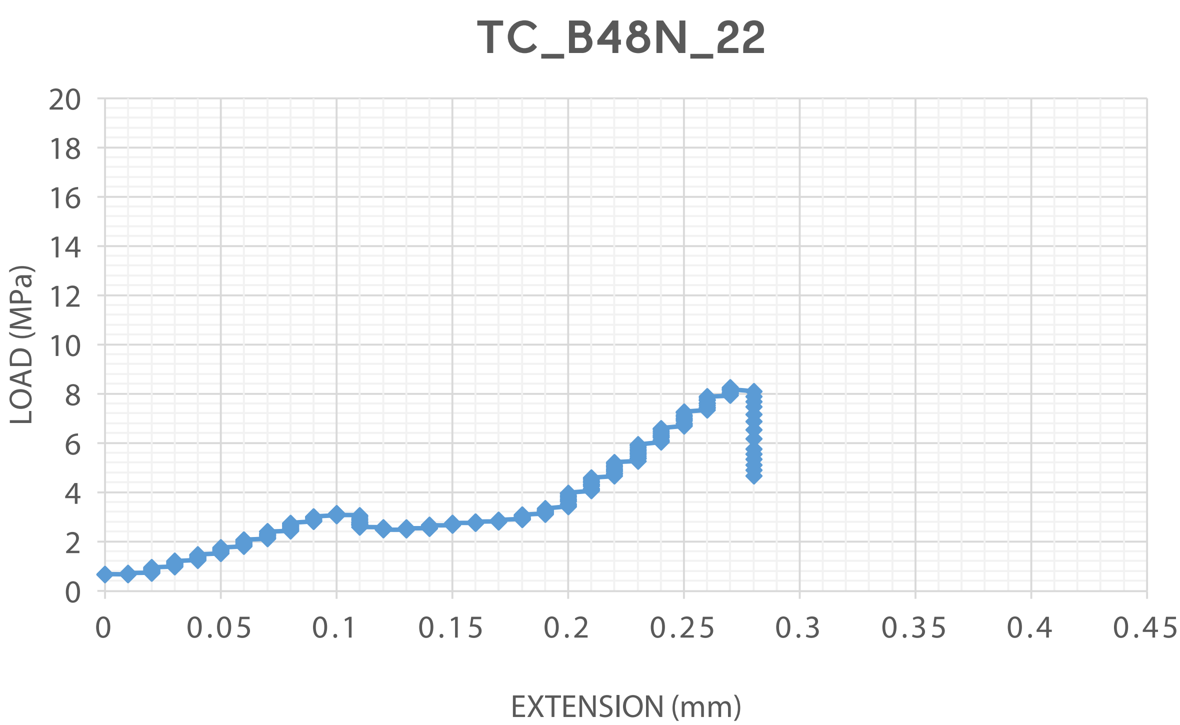
TC_B48N_22
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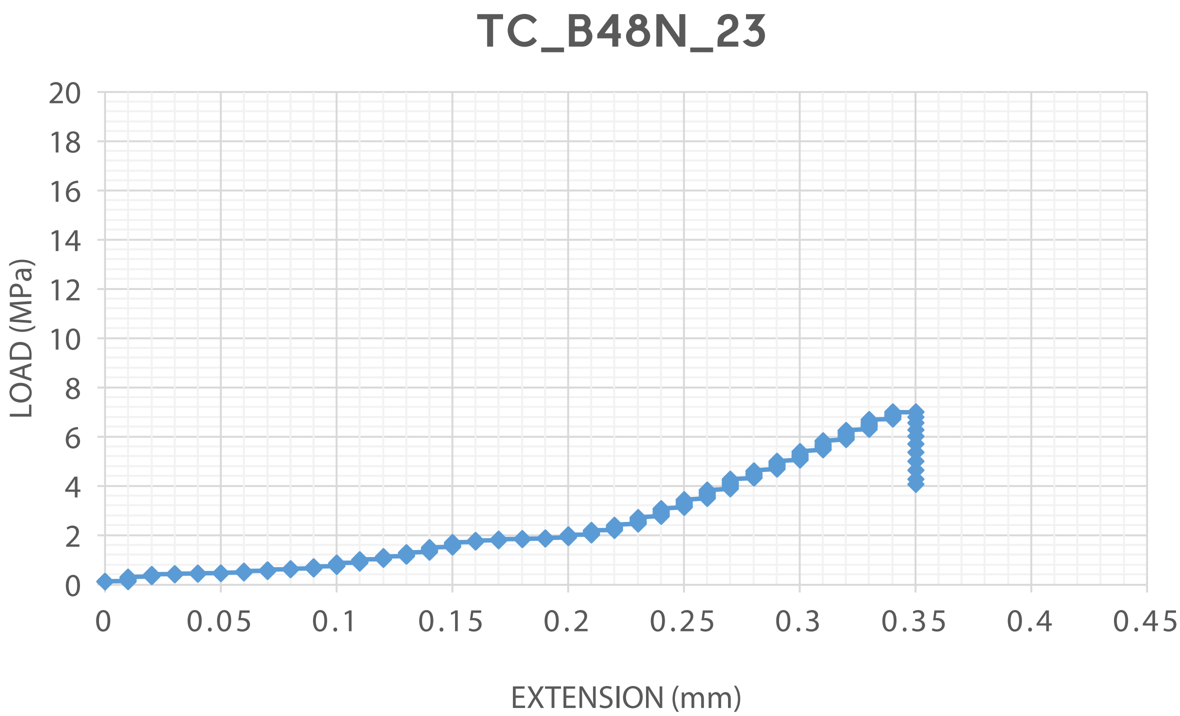
TC_B48N_23
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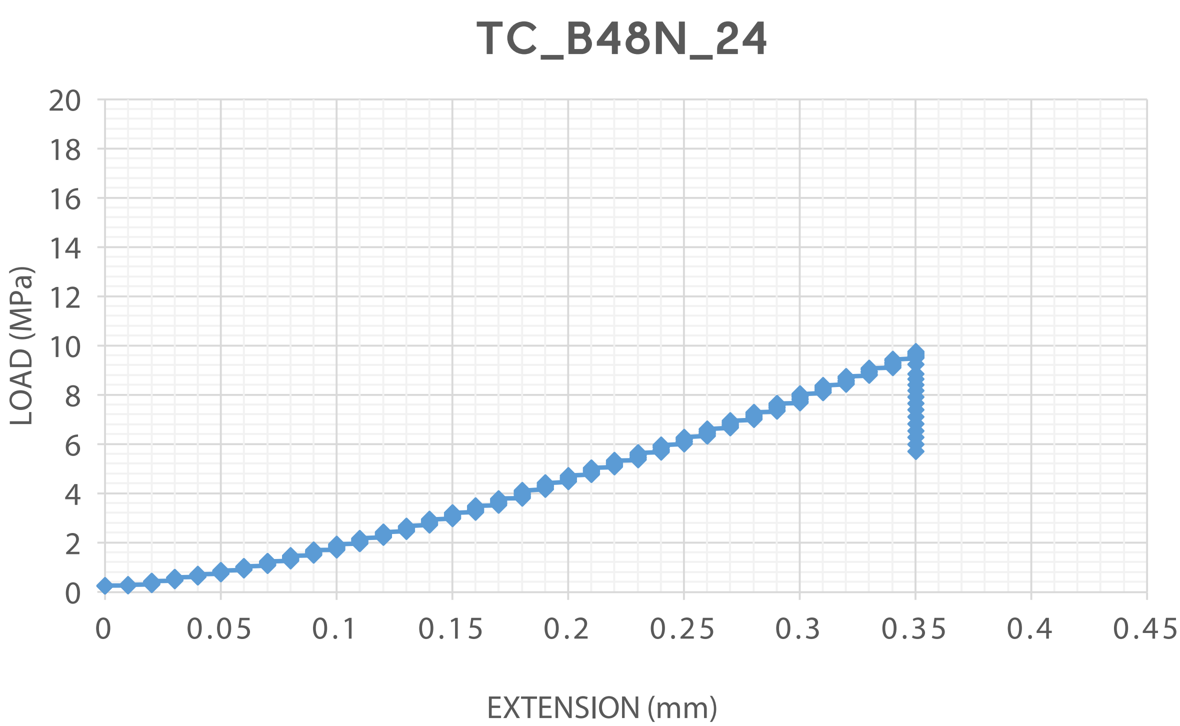
EXTENSION (mm)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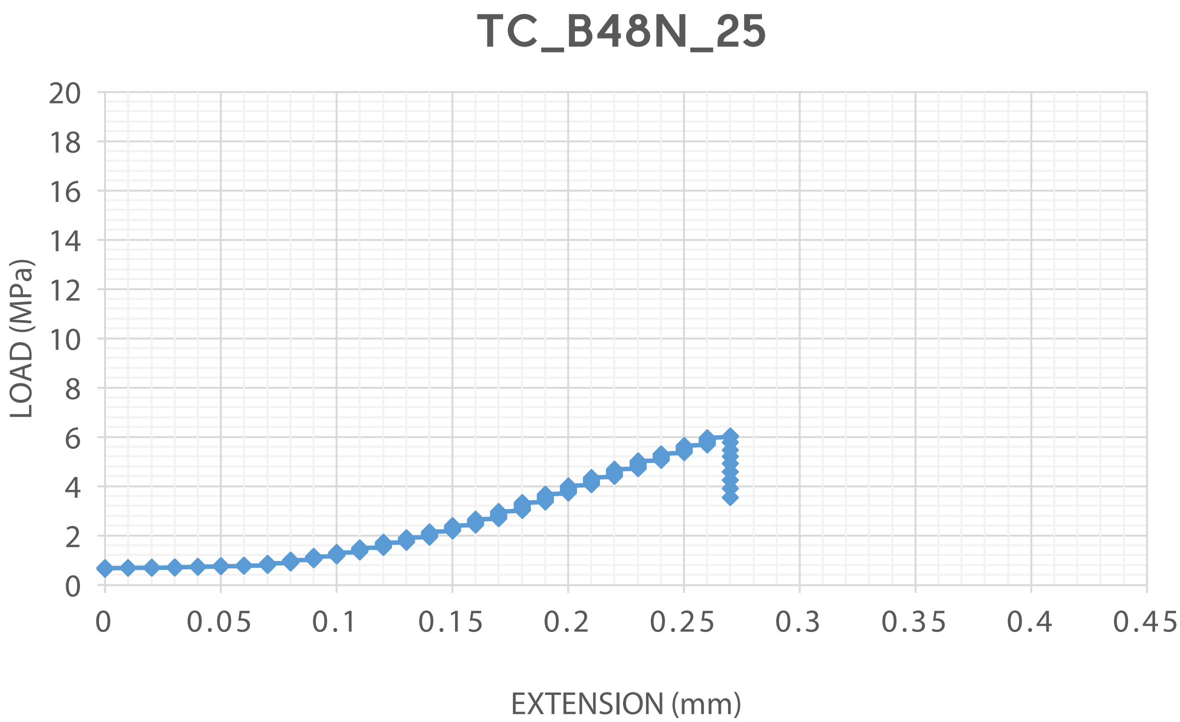
EXTENSION (mm)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EXTENSION (mm)
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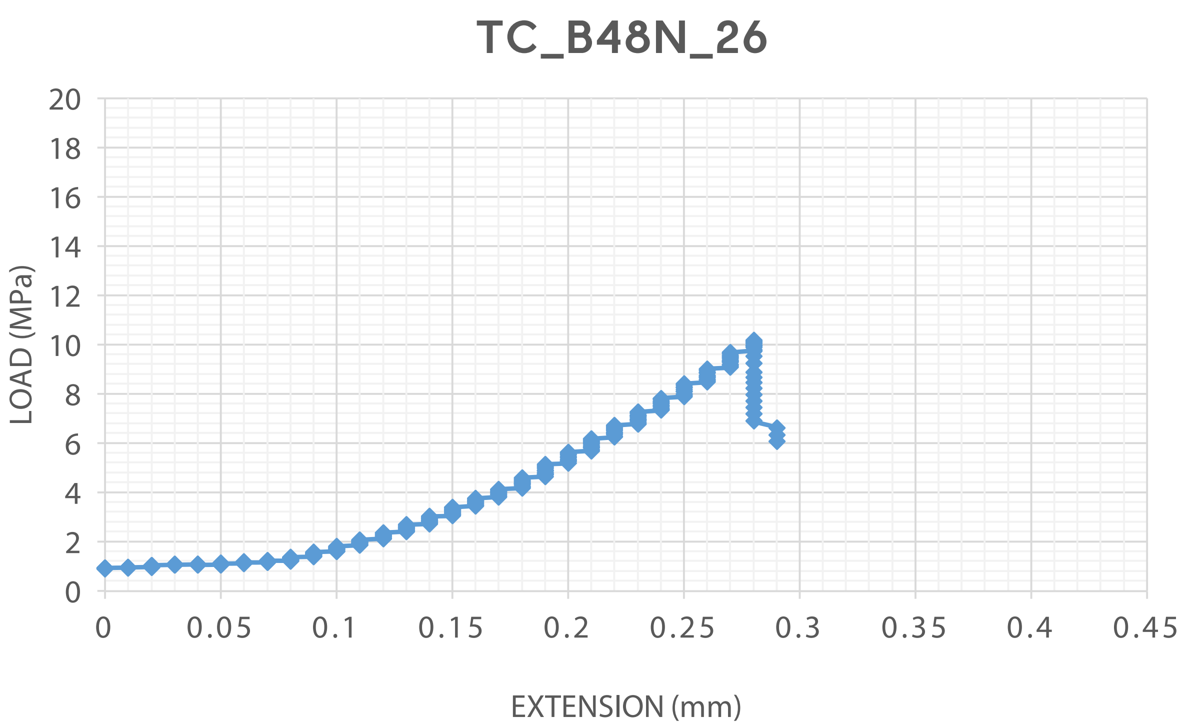
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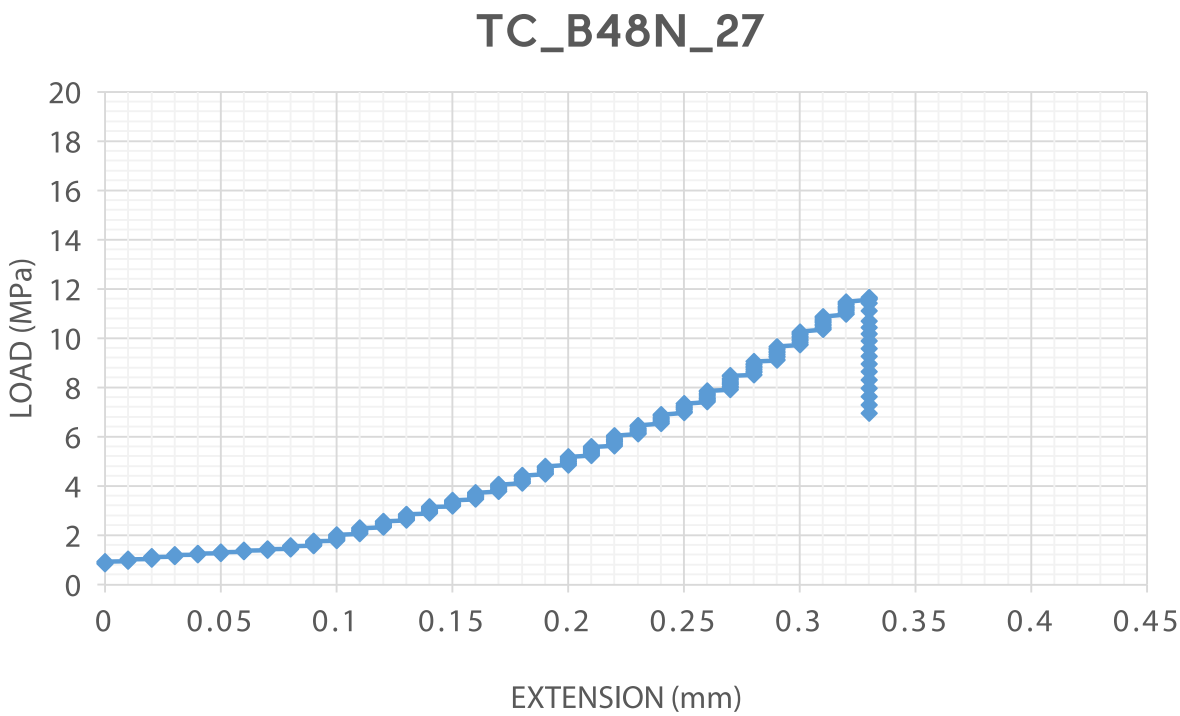
TC_B48N_27
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EXTENSION (mm)
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0.25
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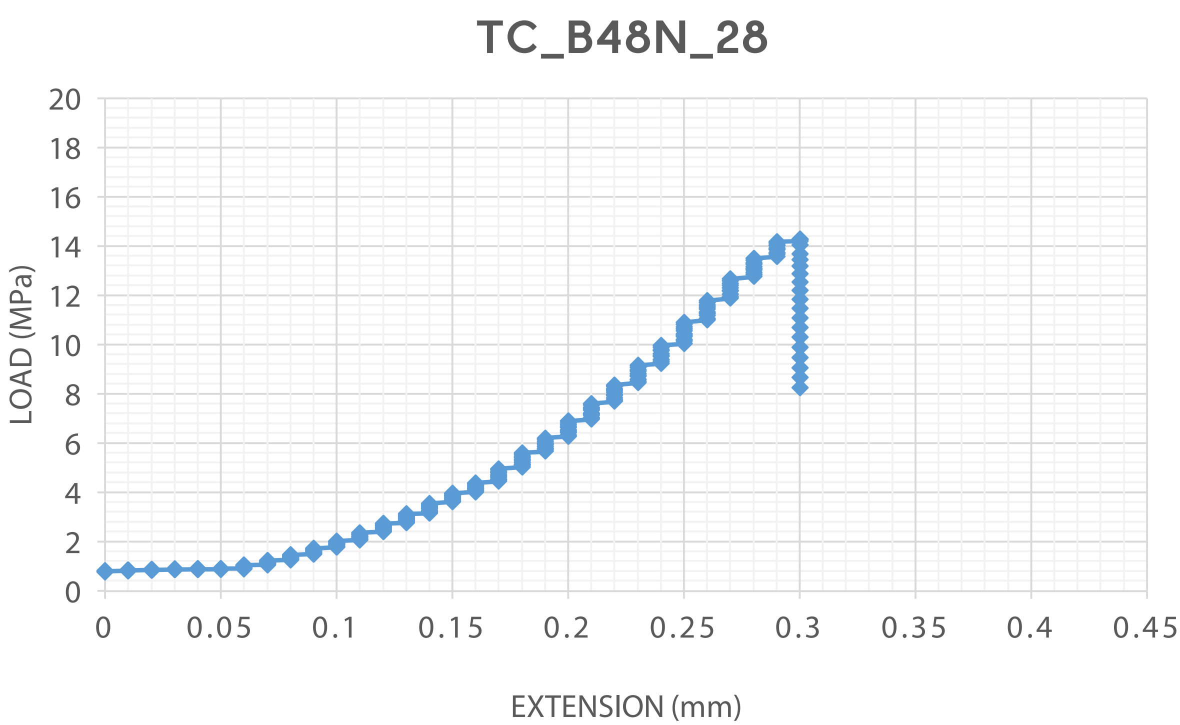
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0.25
0.3
0.35
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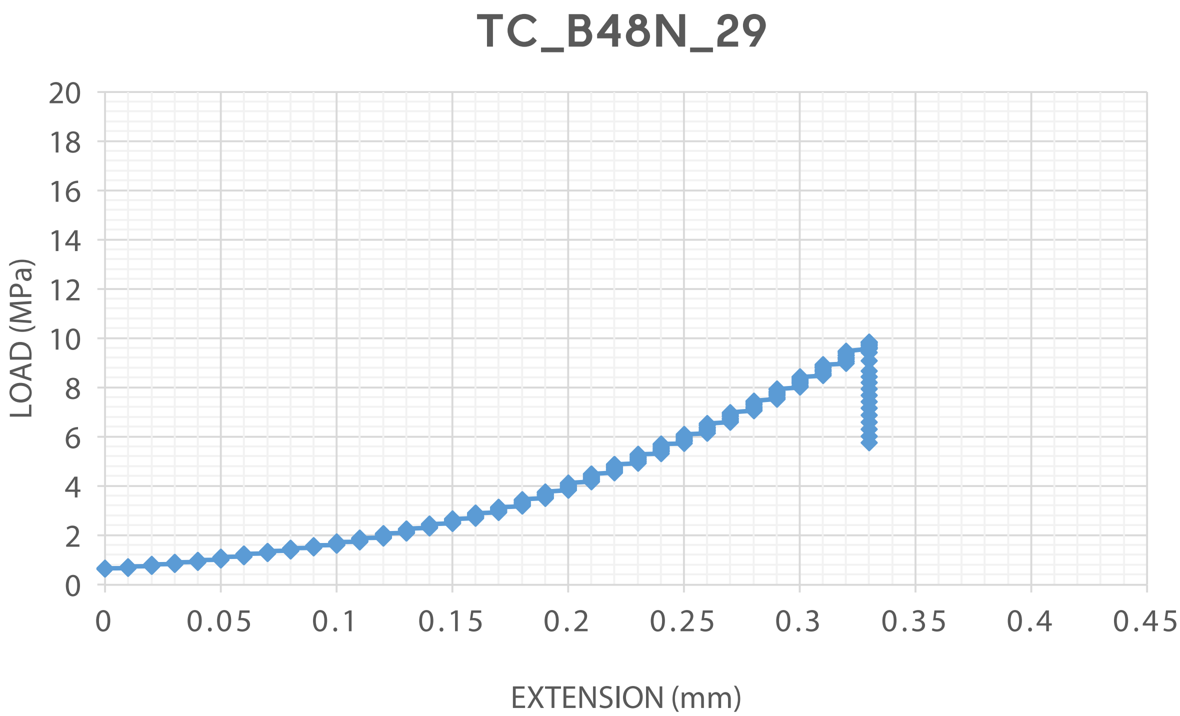
0.45


TC_B48N_29
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EXTENSION (mm)
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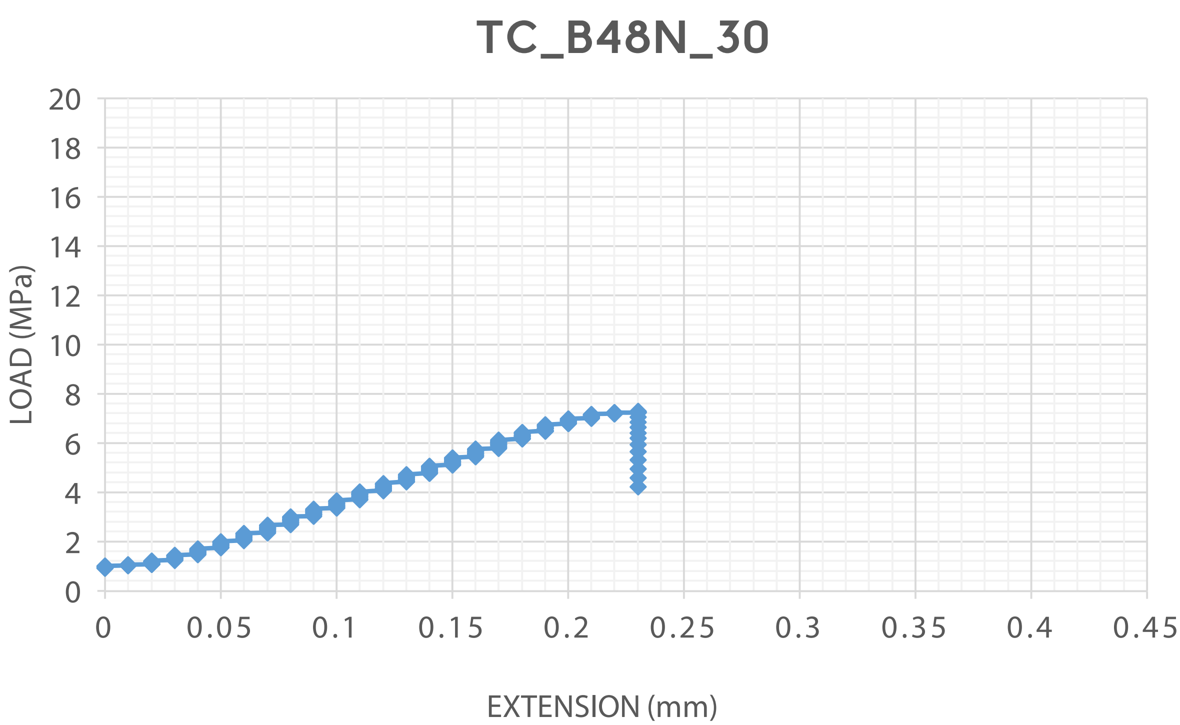
TC_B48N_30
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0.15
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0.25
0.3
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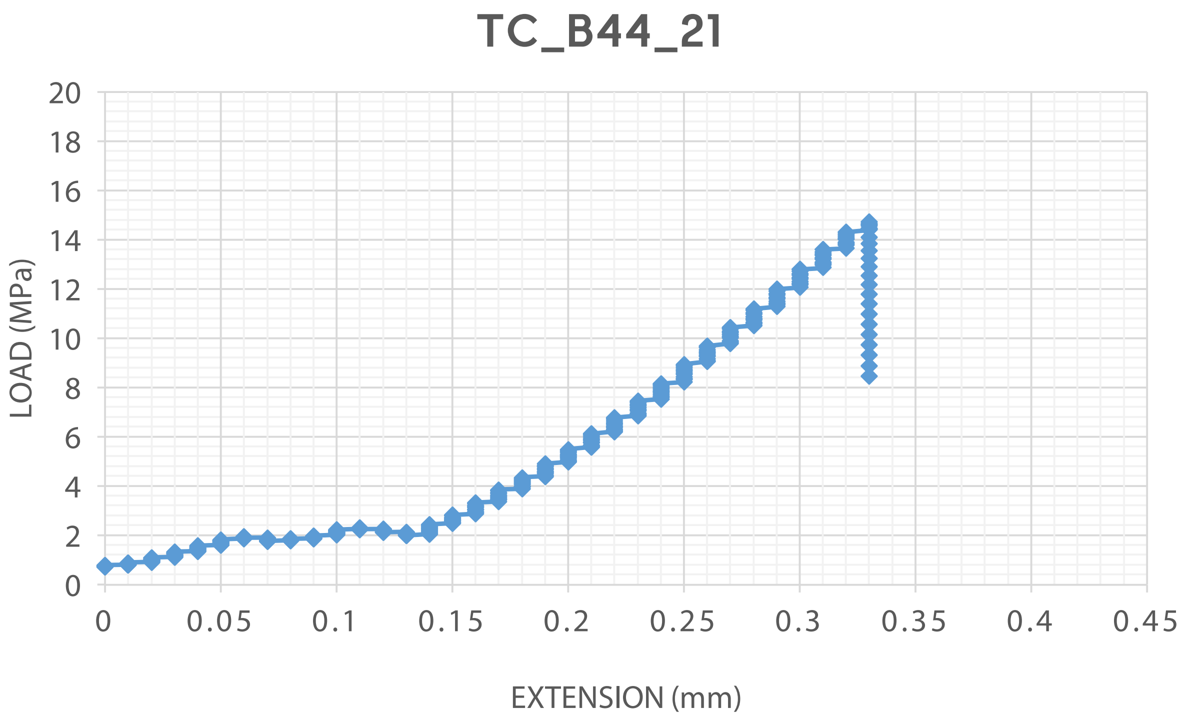
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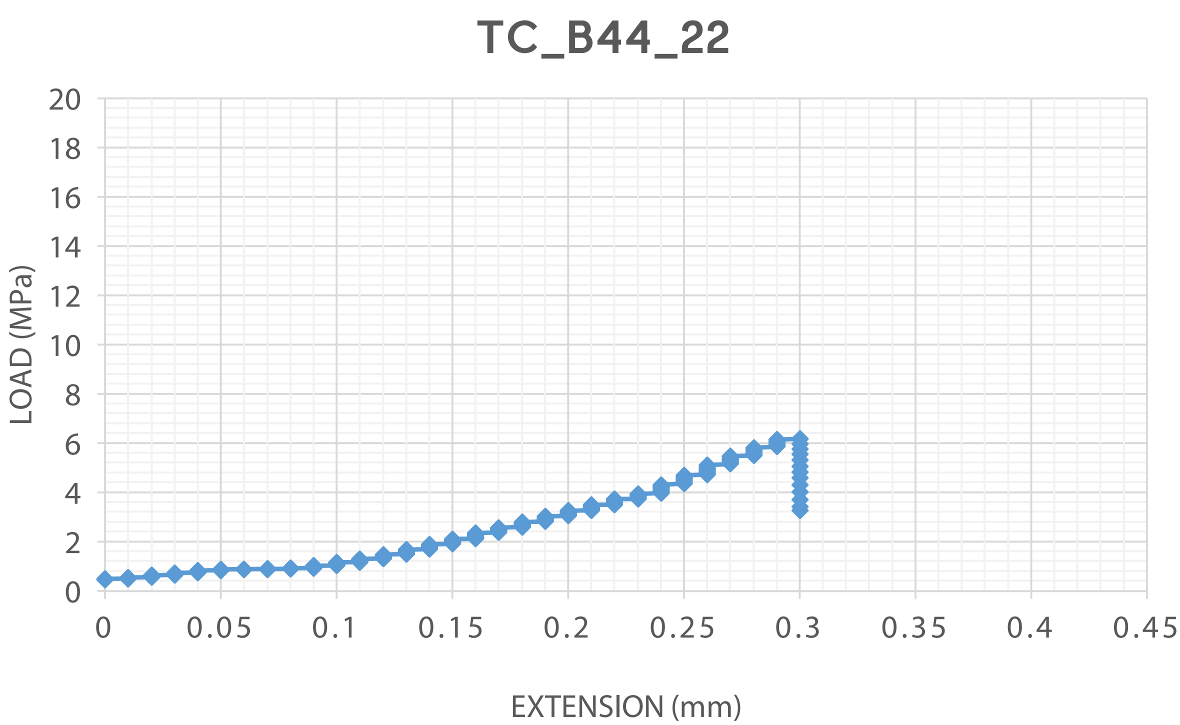
0.45
EXTENSION (mm)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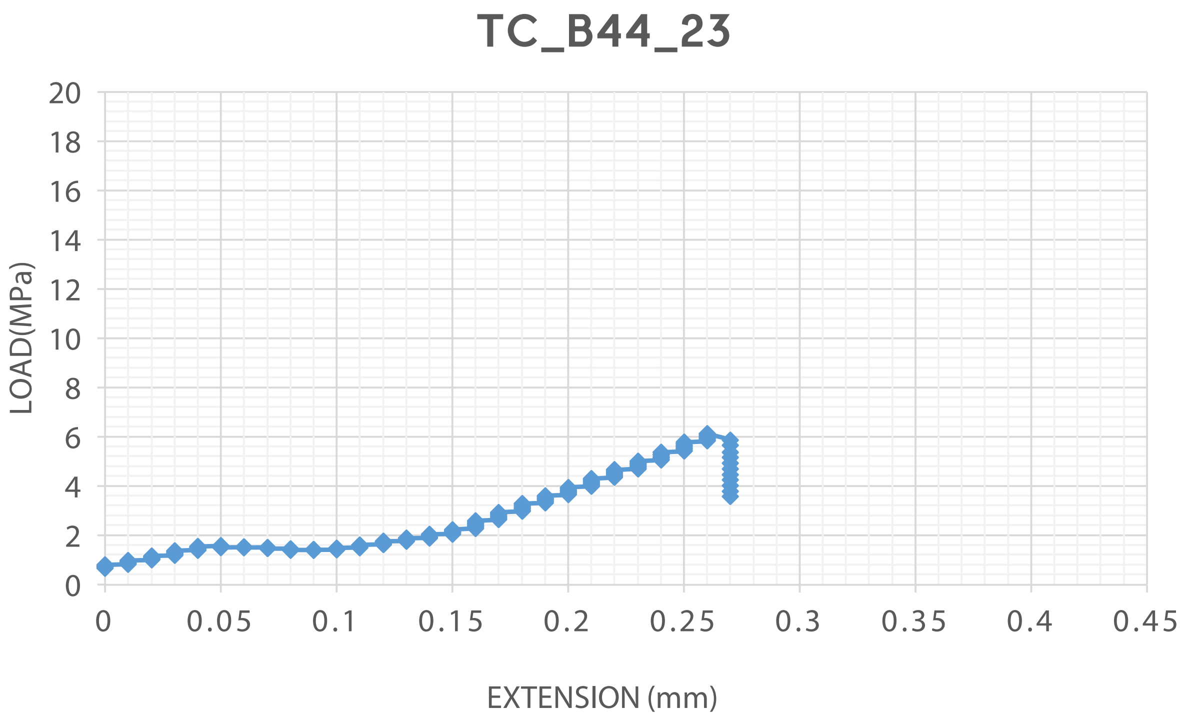
EXTENSION (mm)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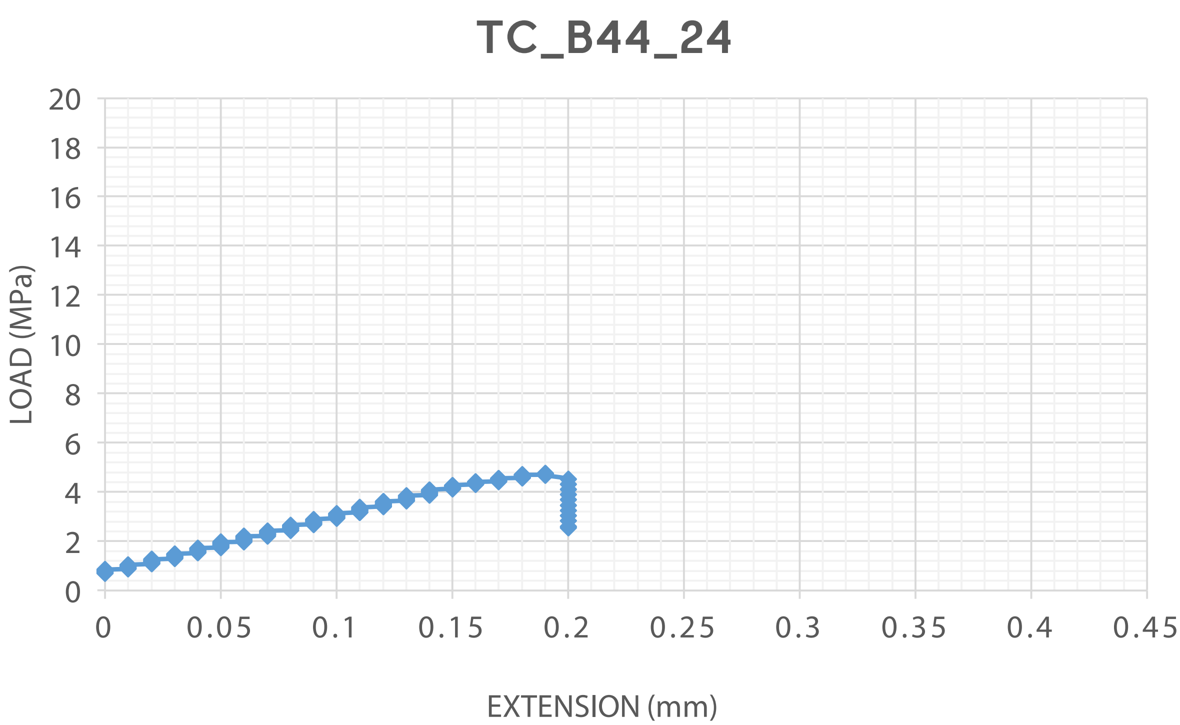
EXTENSION (mm)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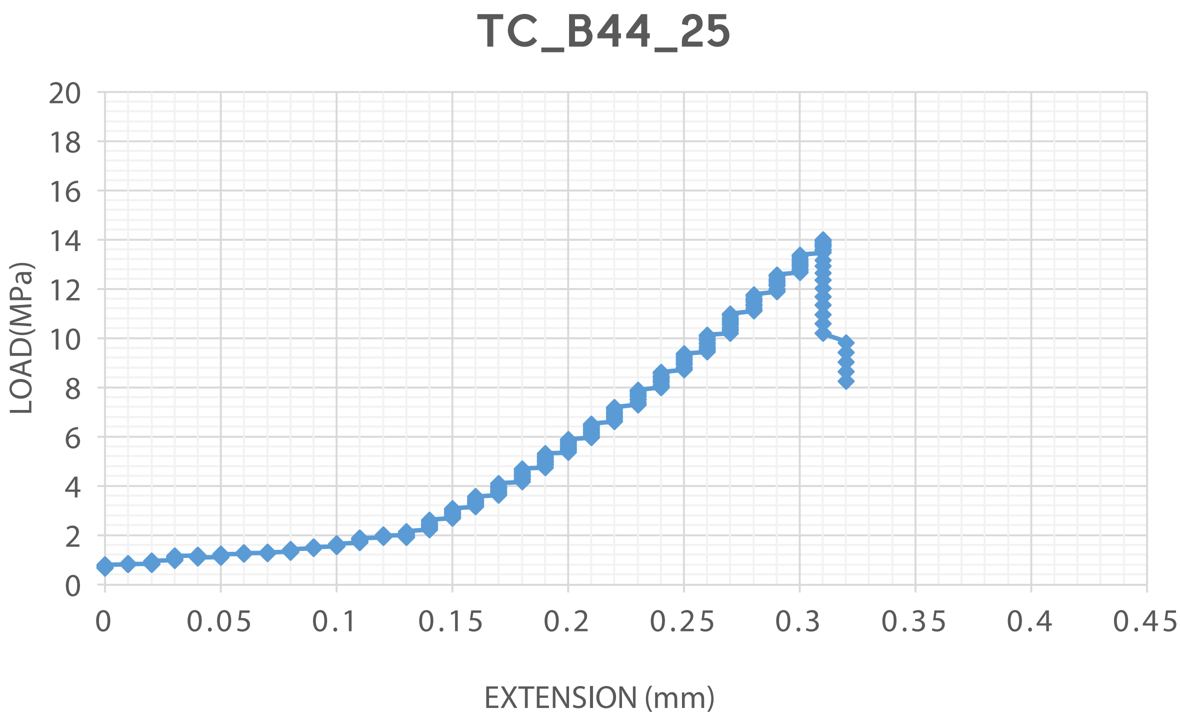
EXTENSION (mm)


TC_B44_25
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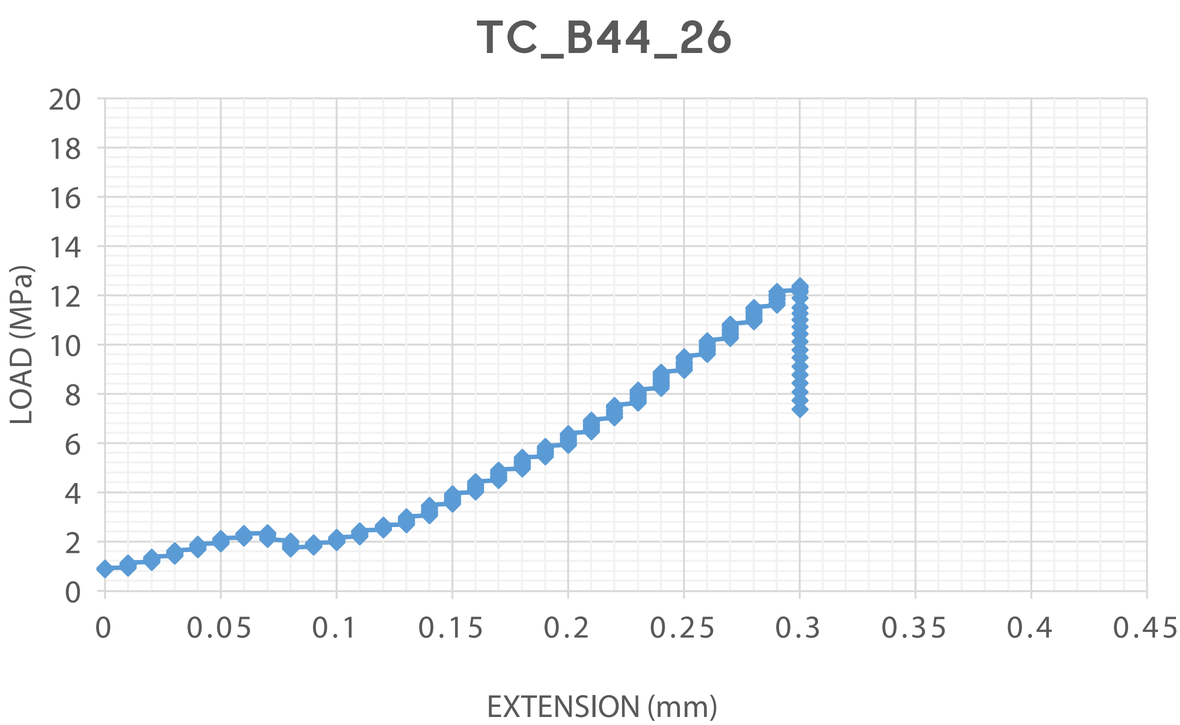
EXTENSION (mm)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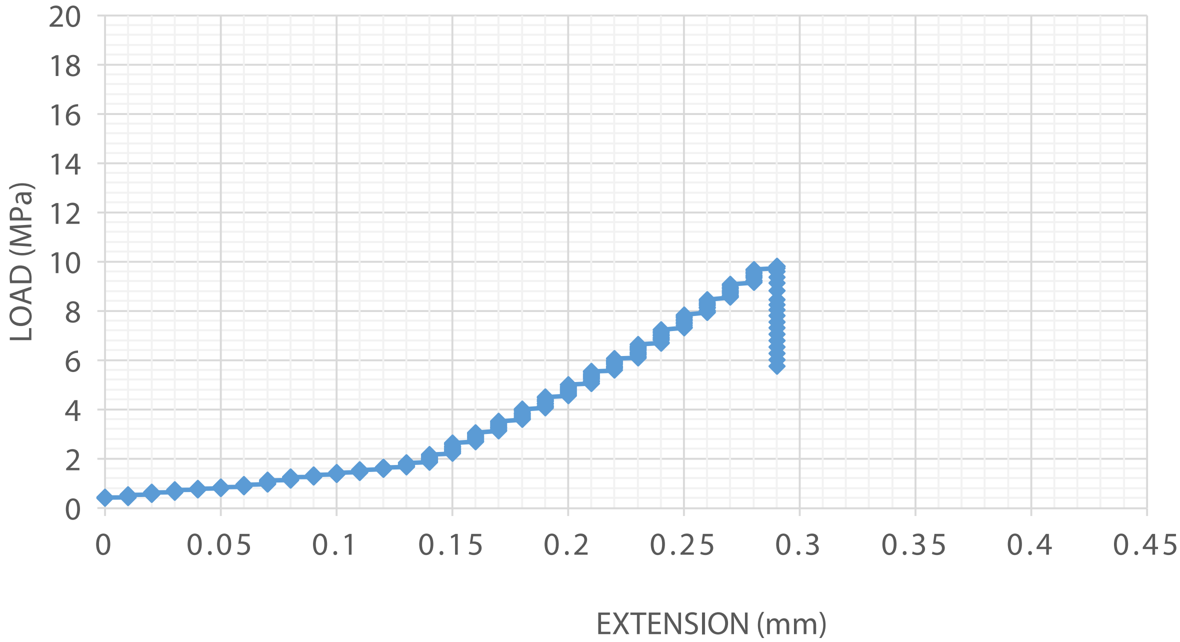
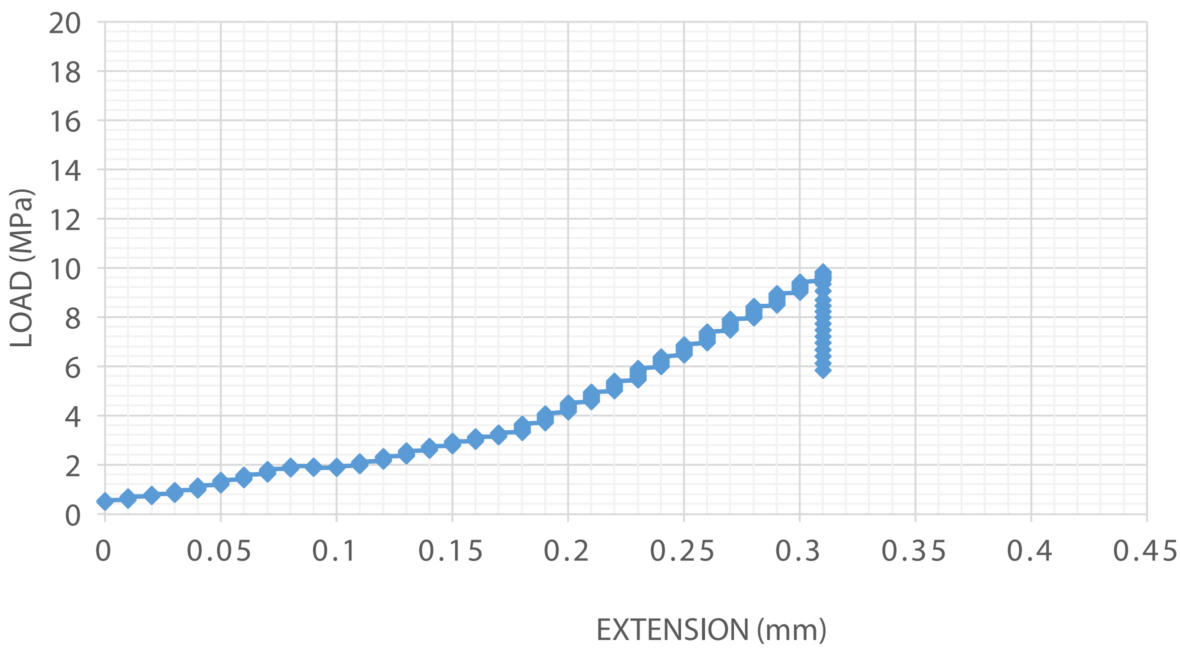
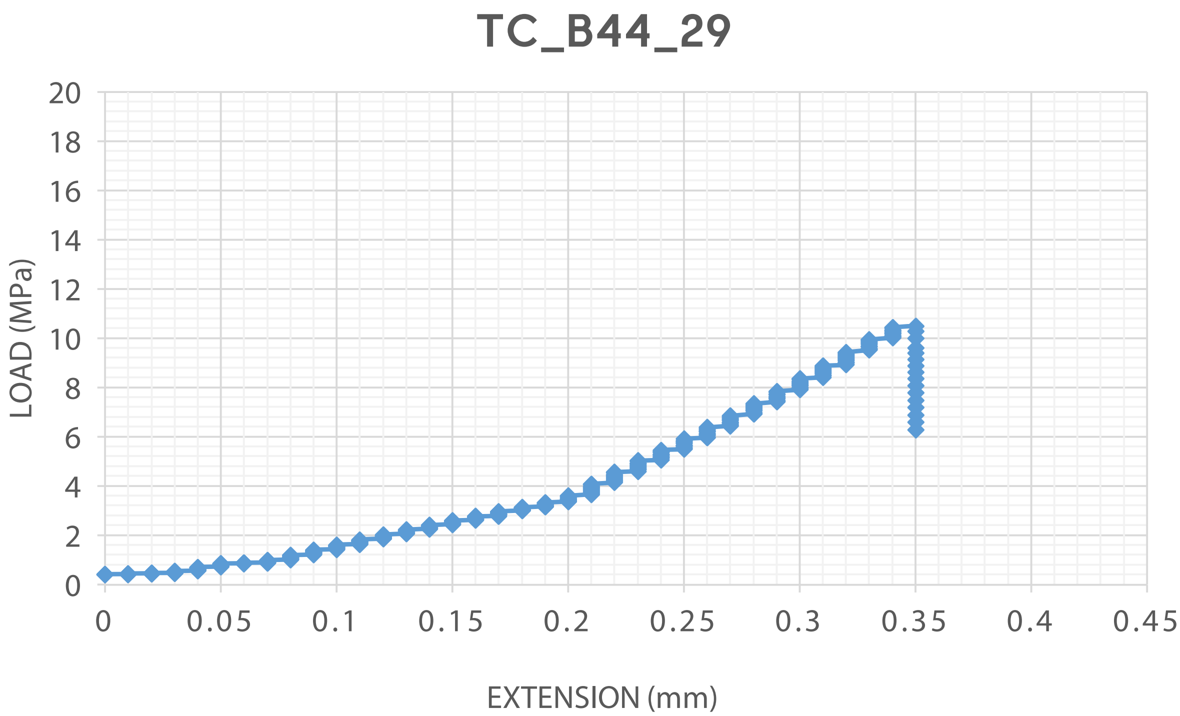
0.45
EXTENSION (mm)

TC_B44_27



TC_B44_28

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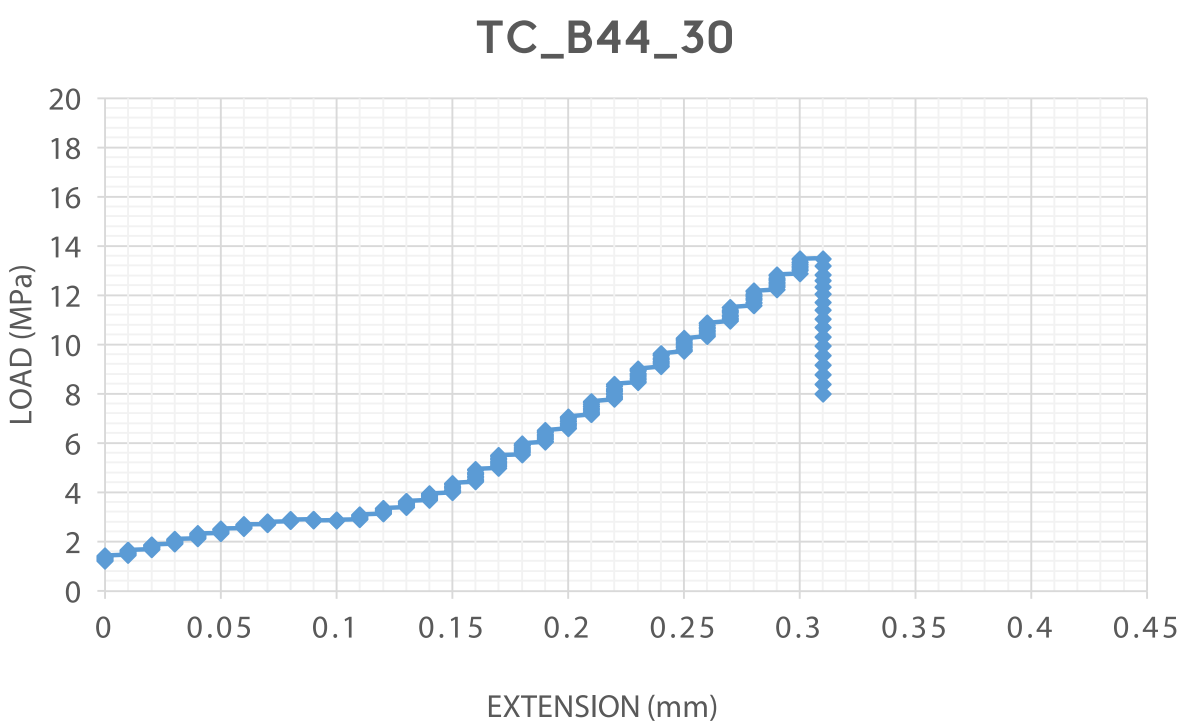
EXTENSION (mm)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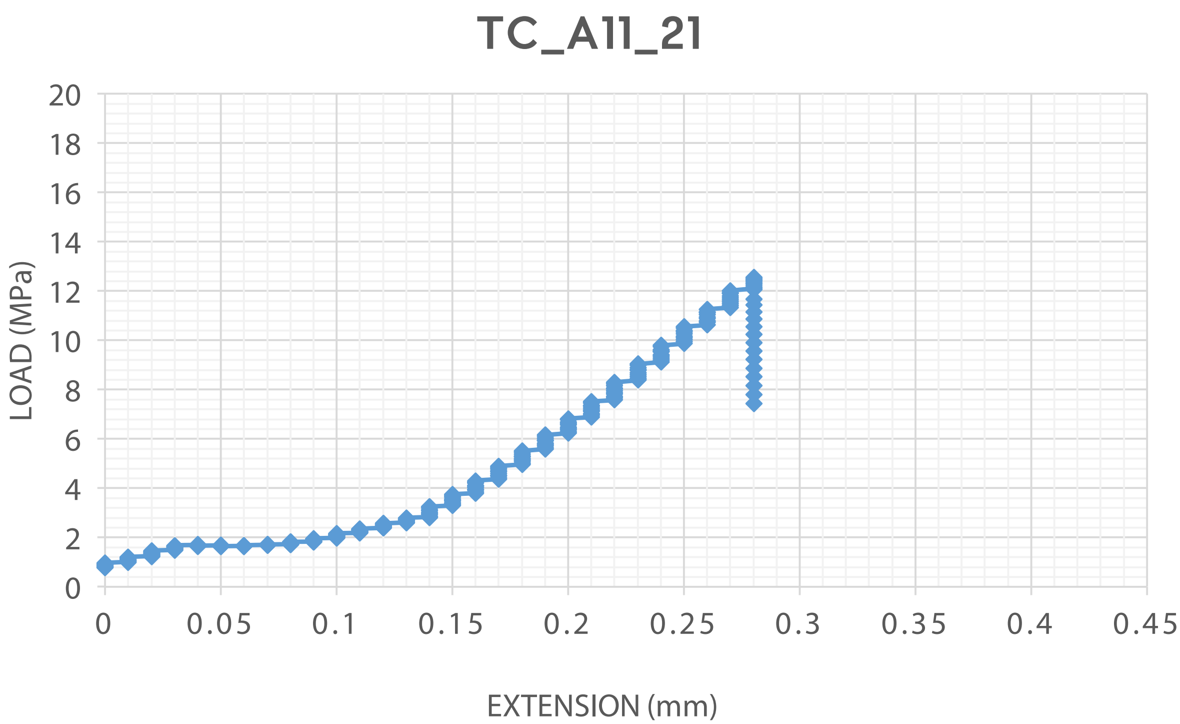
0.45
EXTENSION (mm)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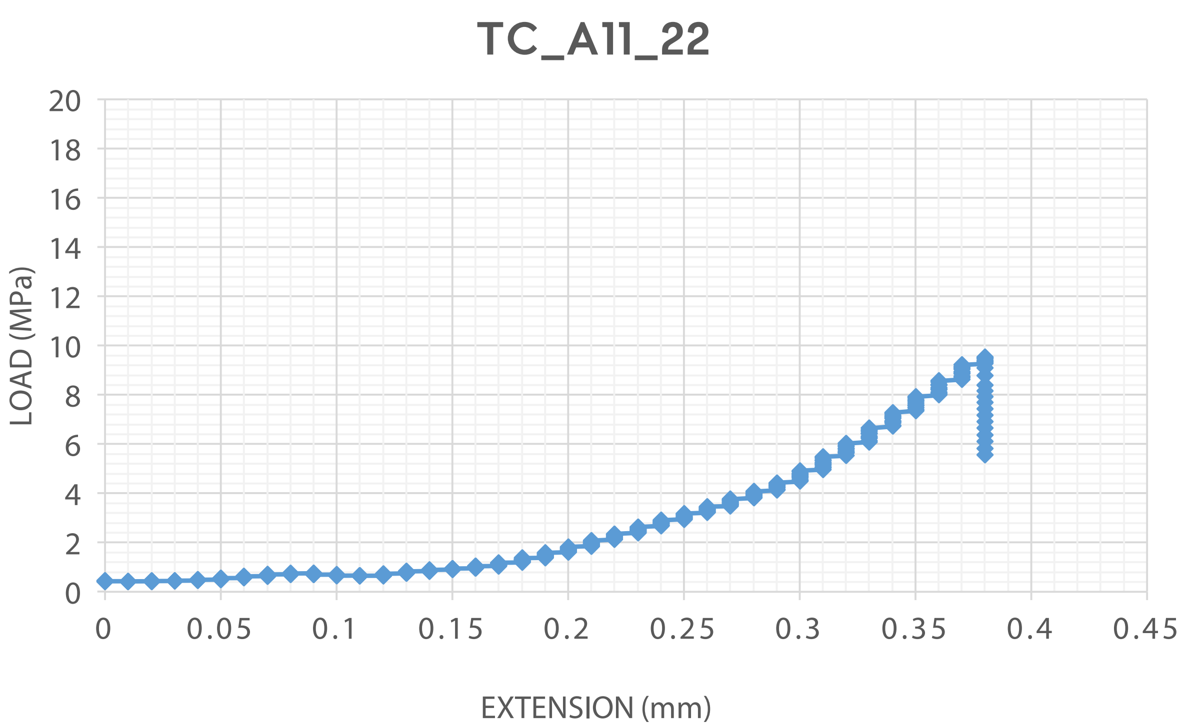
EXTENSION (mm)


TC_A11_22
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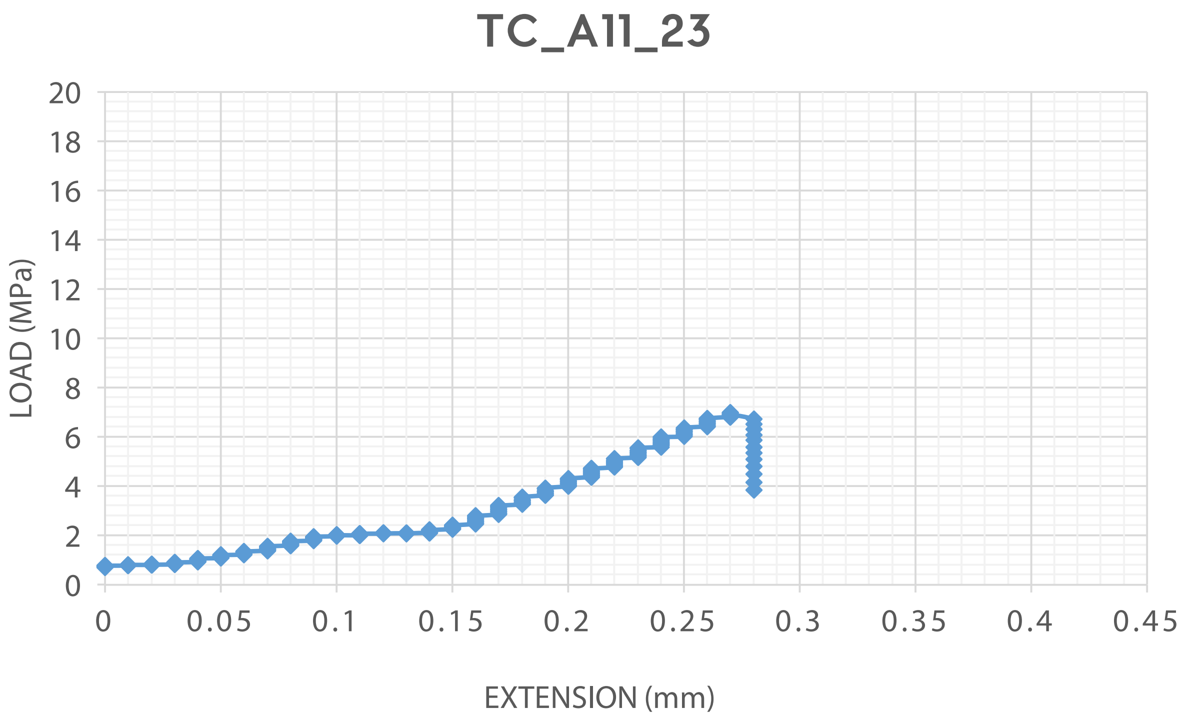
EXTENSION (mm)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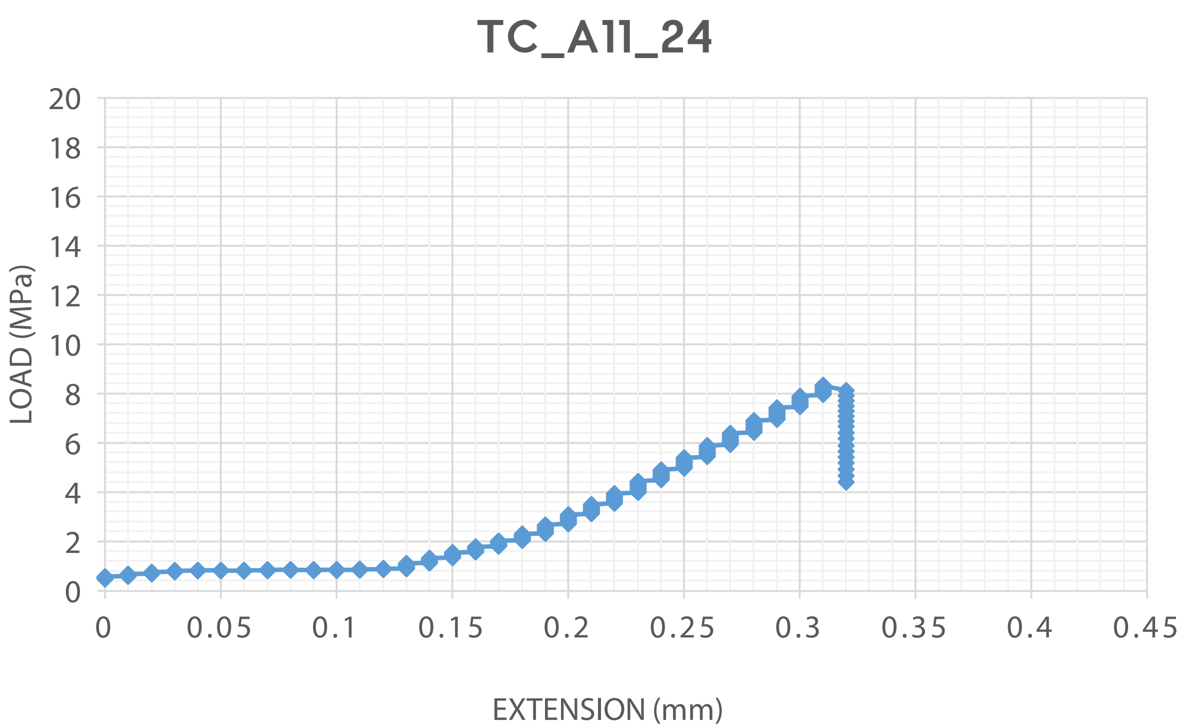
TC_A11_24
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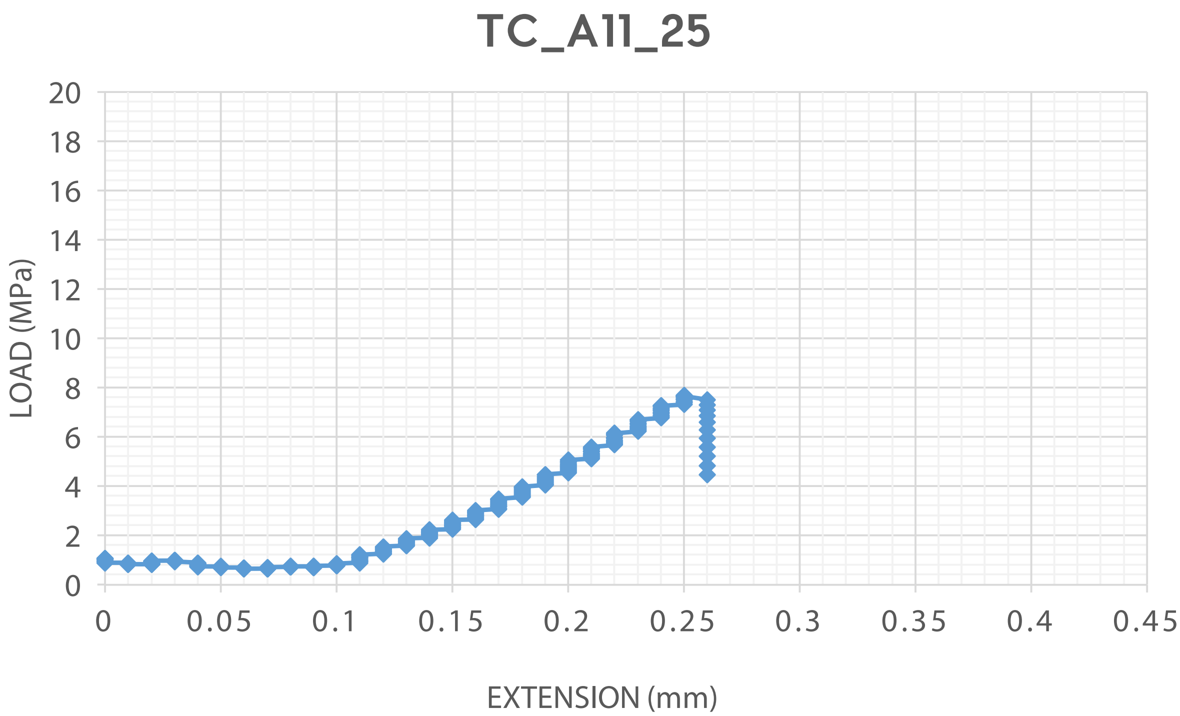
EXTENSION (mm)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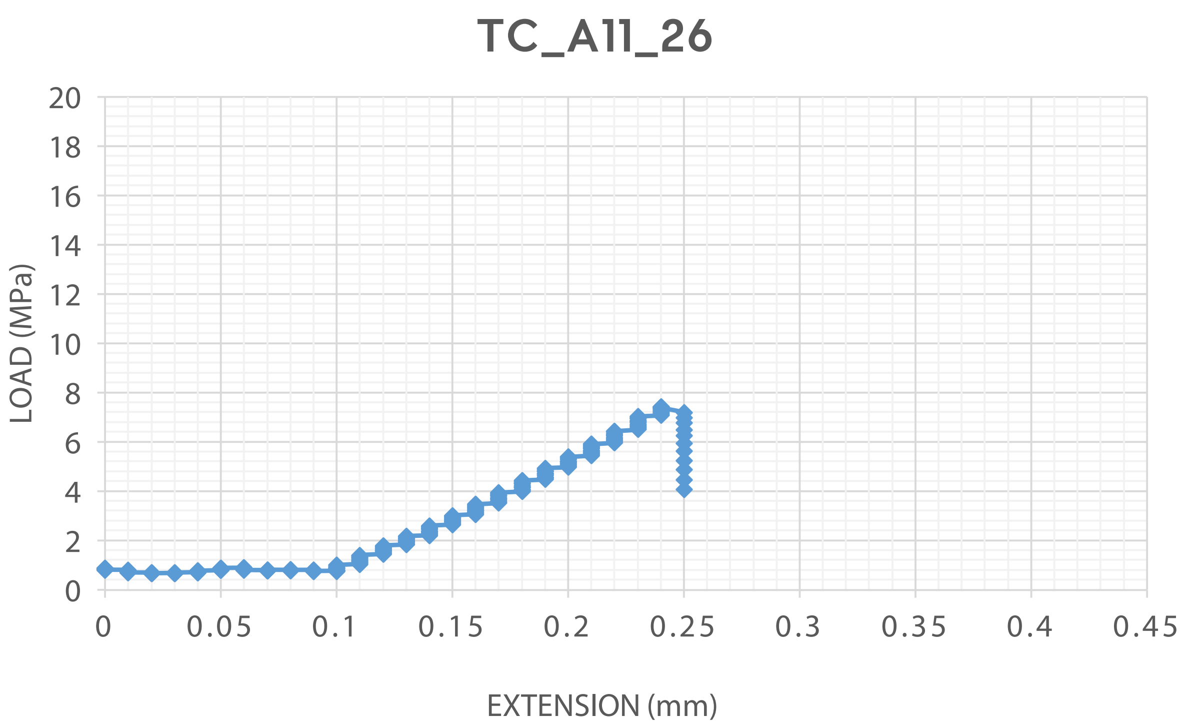
EXTENSION (mm)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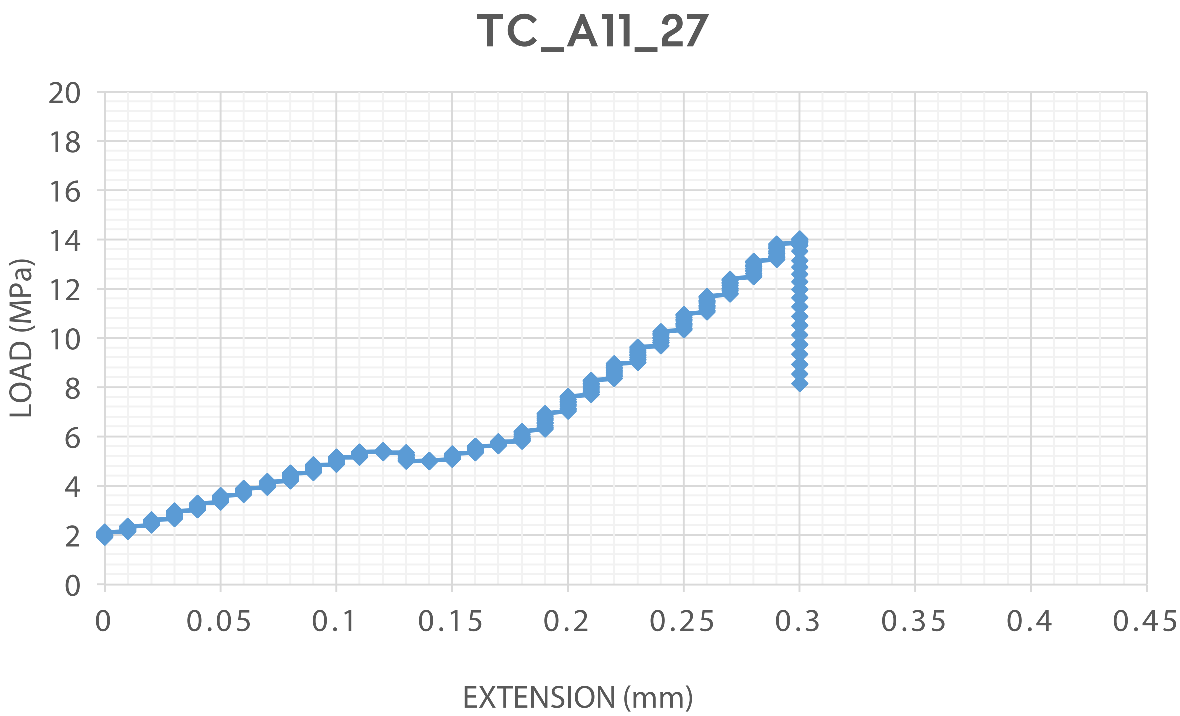
EXTENSION (mm)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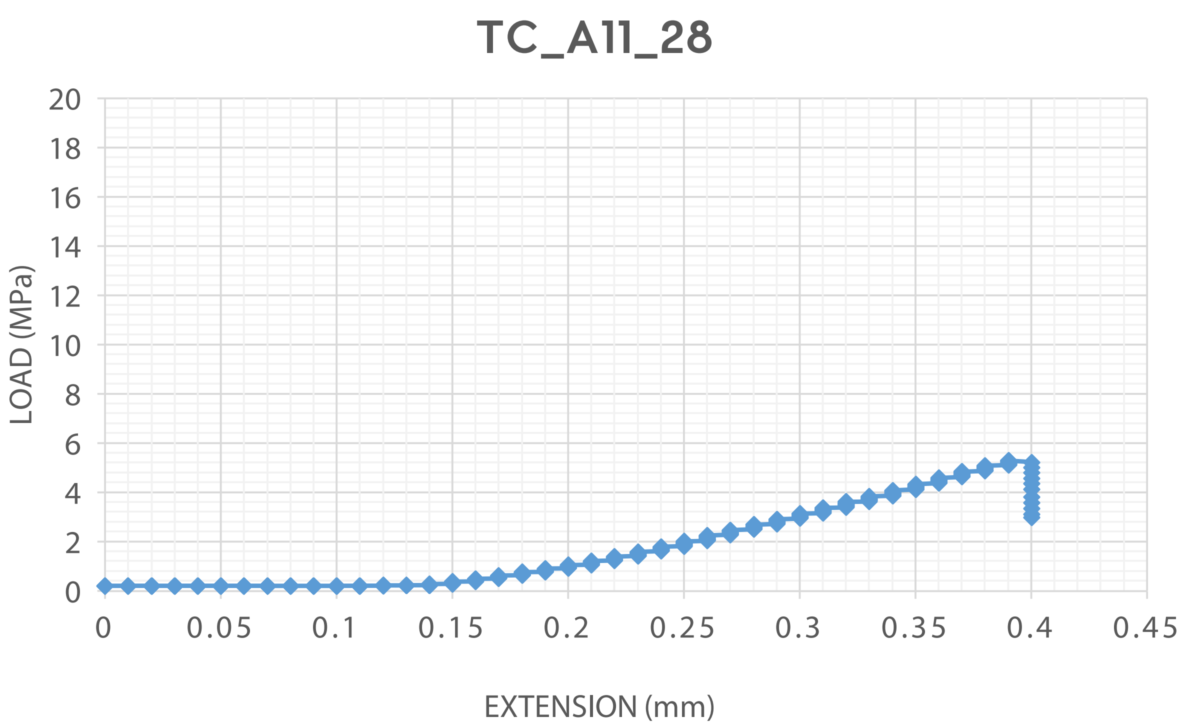
EXTENSION (mm)


TC_A11_28
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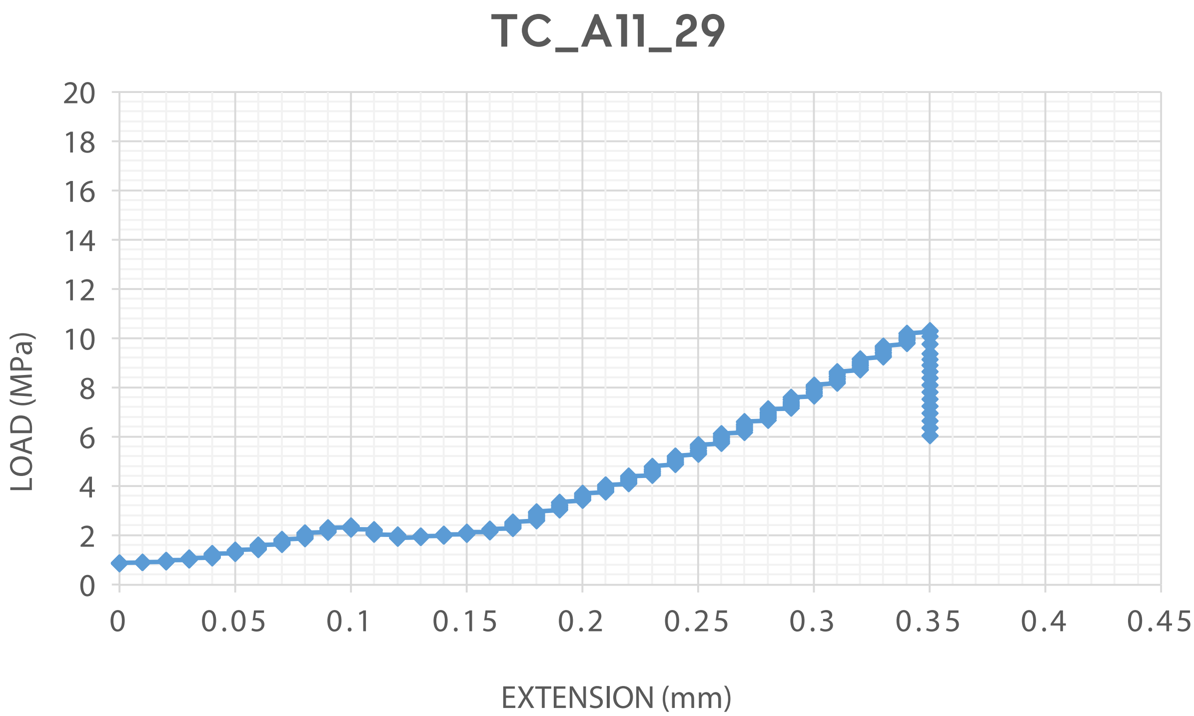
EXTENSION (mm)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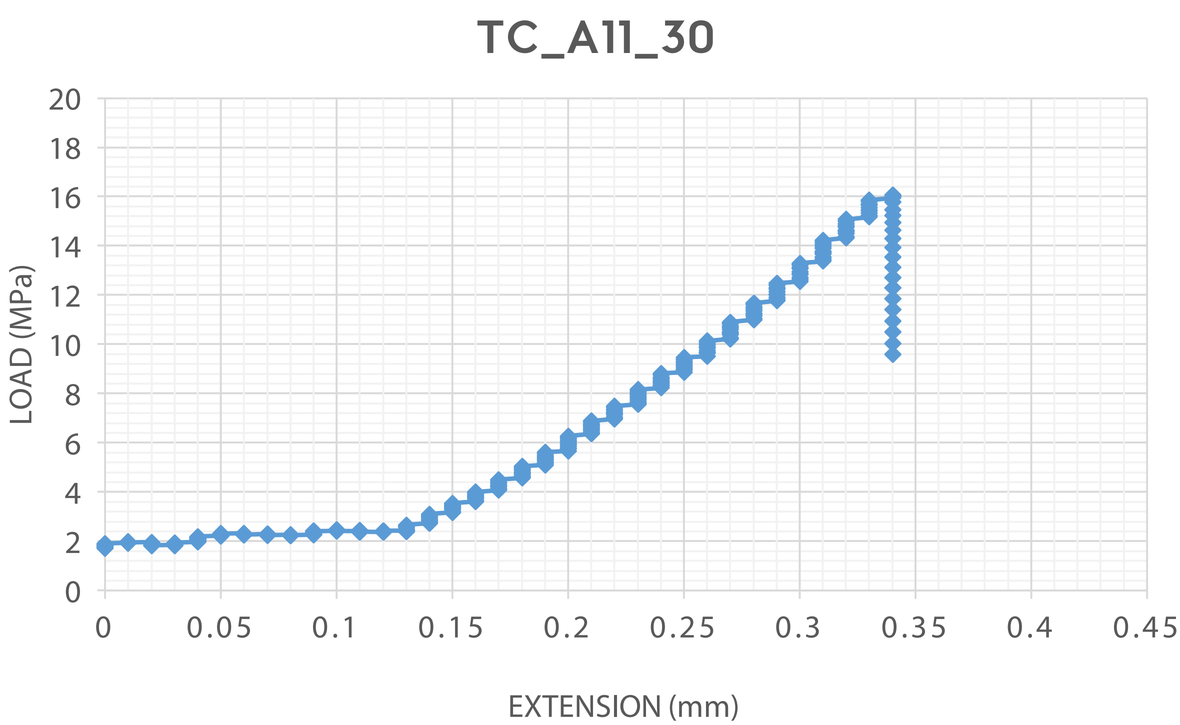
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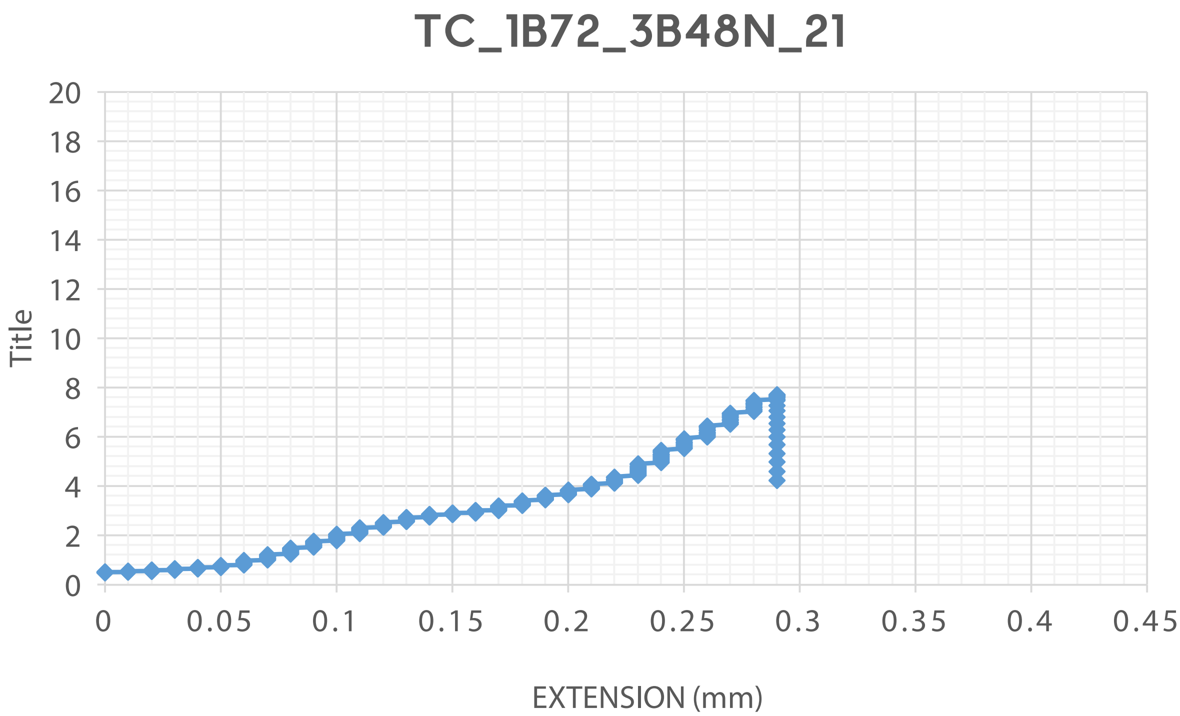
TC_1B72_3B48N_21
Title
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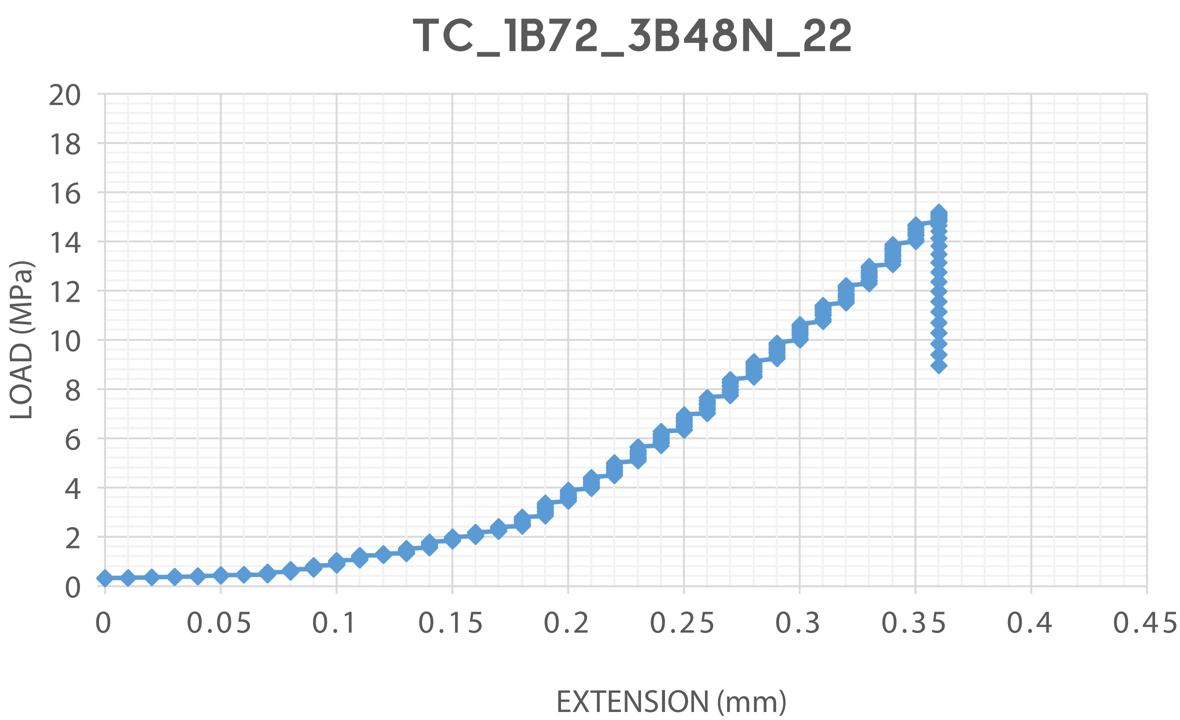
TC_1B72_3B48N_22
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EXTENSION (mm)
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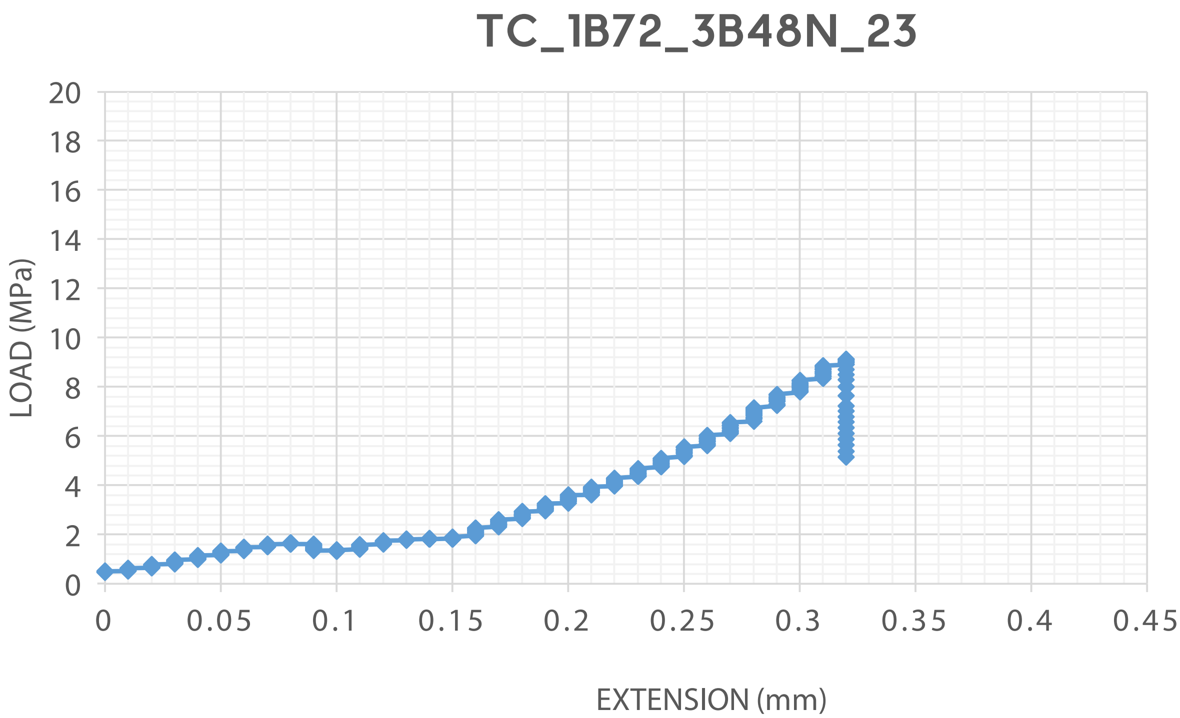
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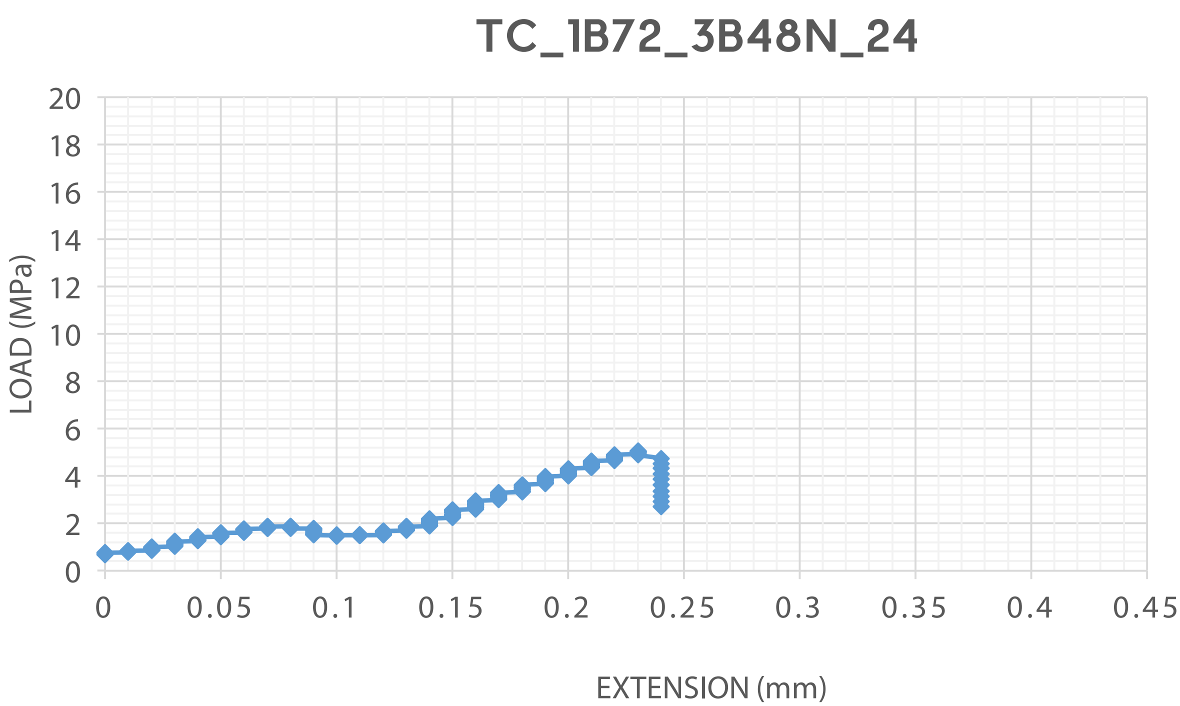
EXTENSION (mm)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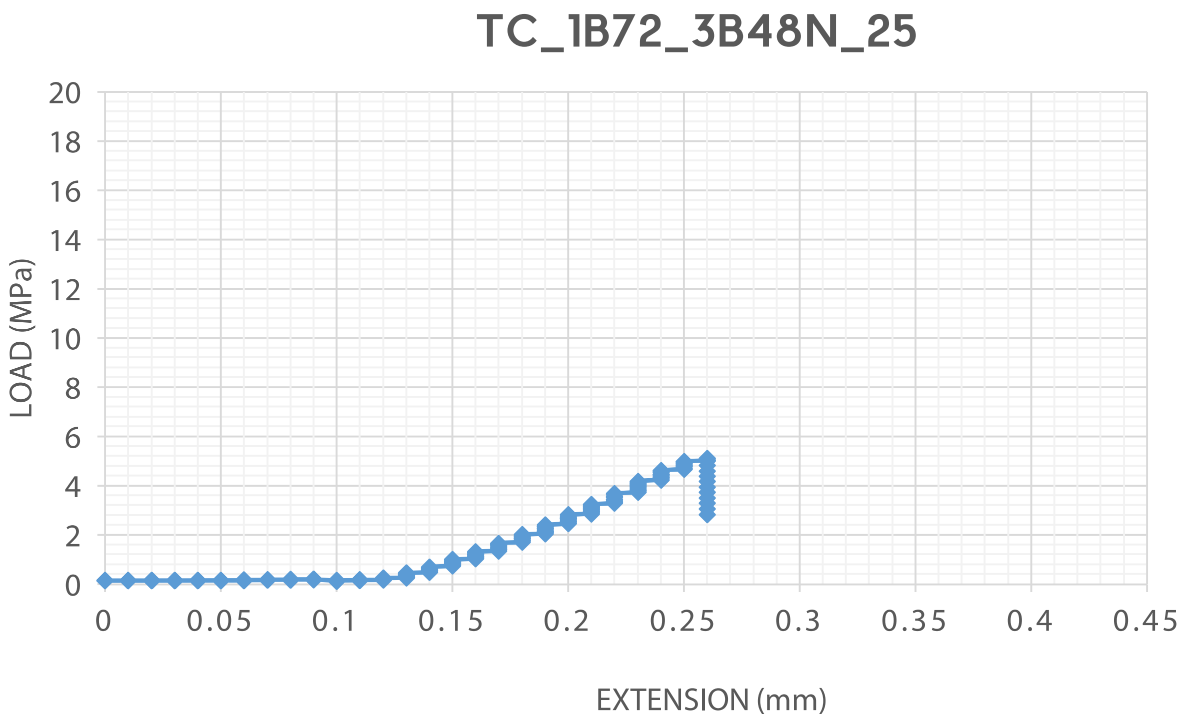
EXTENSION (mm)


TC_1B72_3B48N_25
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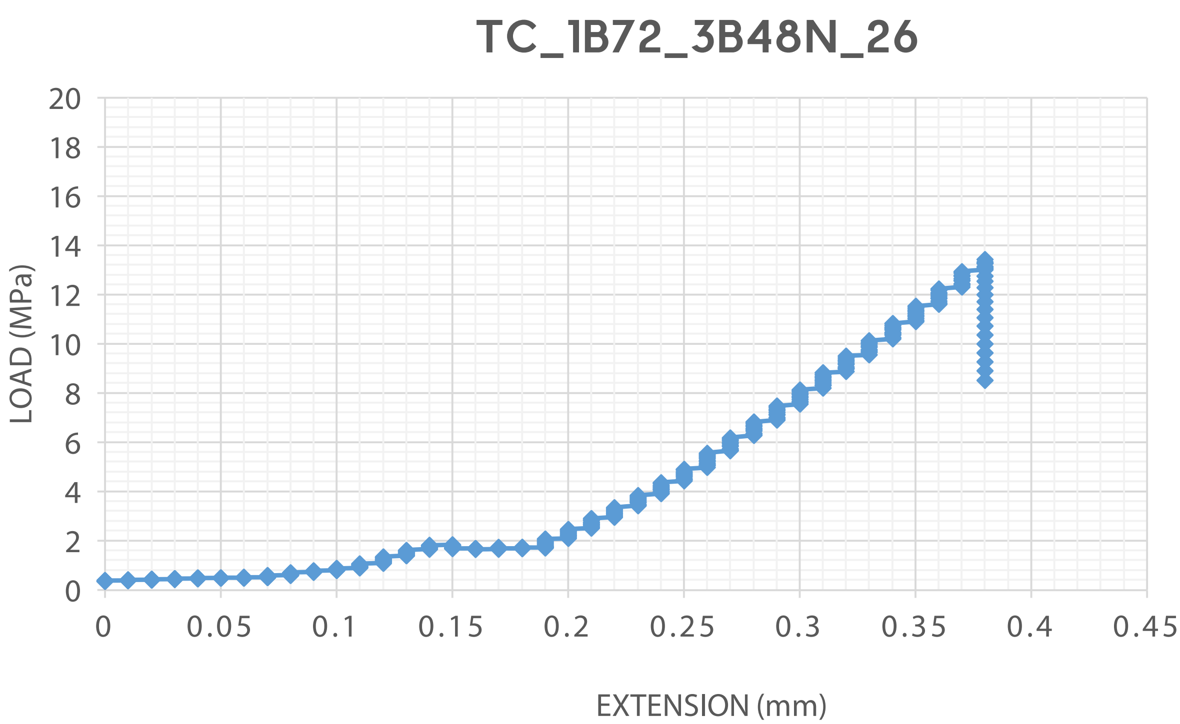
TC_1B72_3B48N_26
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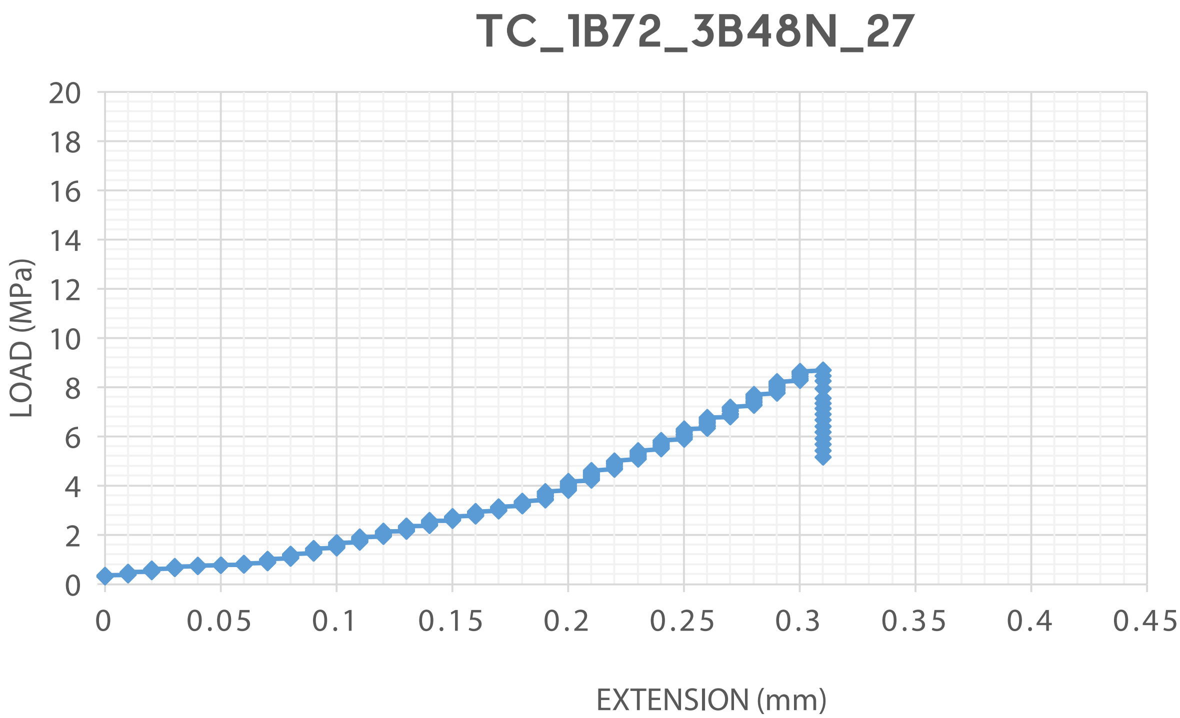
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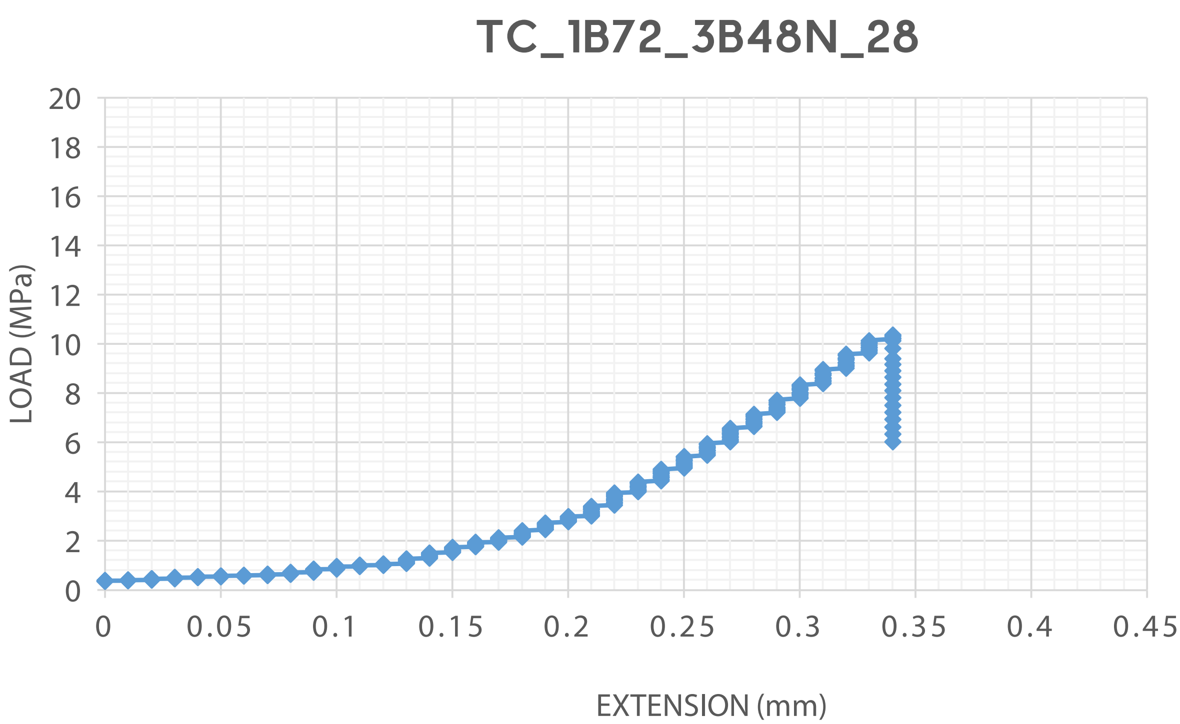
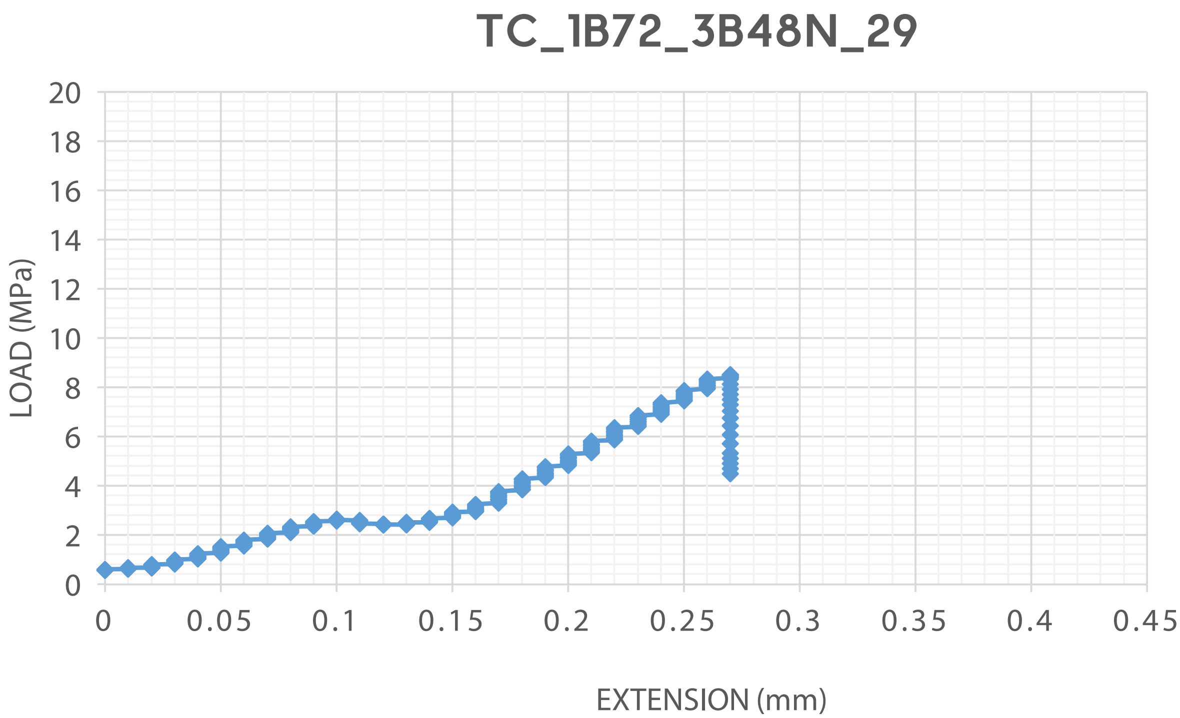
0.4
0.45


TC_1B72_3B48N_28
LOAD (MPa)
EXTENSION (mm)
0
2
4
6
8
10
12
14
16
18
20
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_1B72_3B48N_29
LOAD (MPa)
EXTENSION (mm)
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4
6
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10
12
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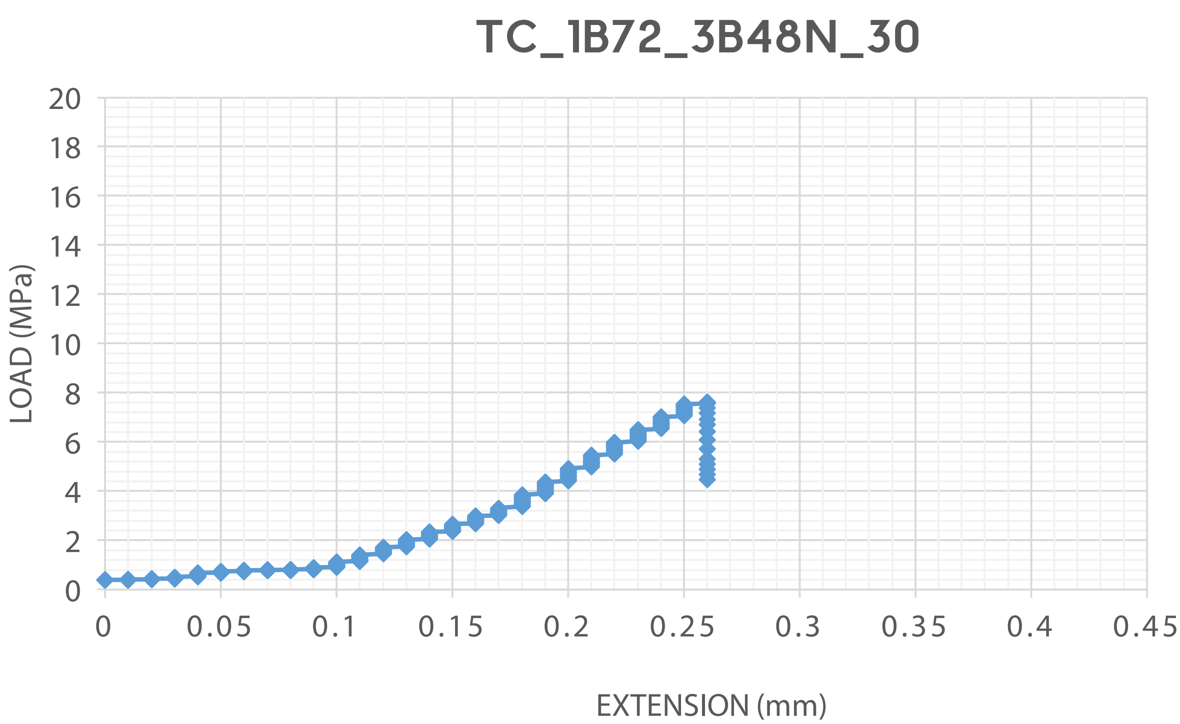
20
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_1B72_3B48N_30
LOAD (MPa)
EXTENSION (mm)
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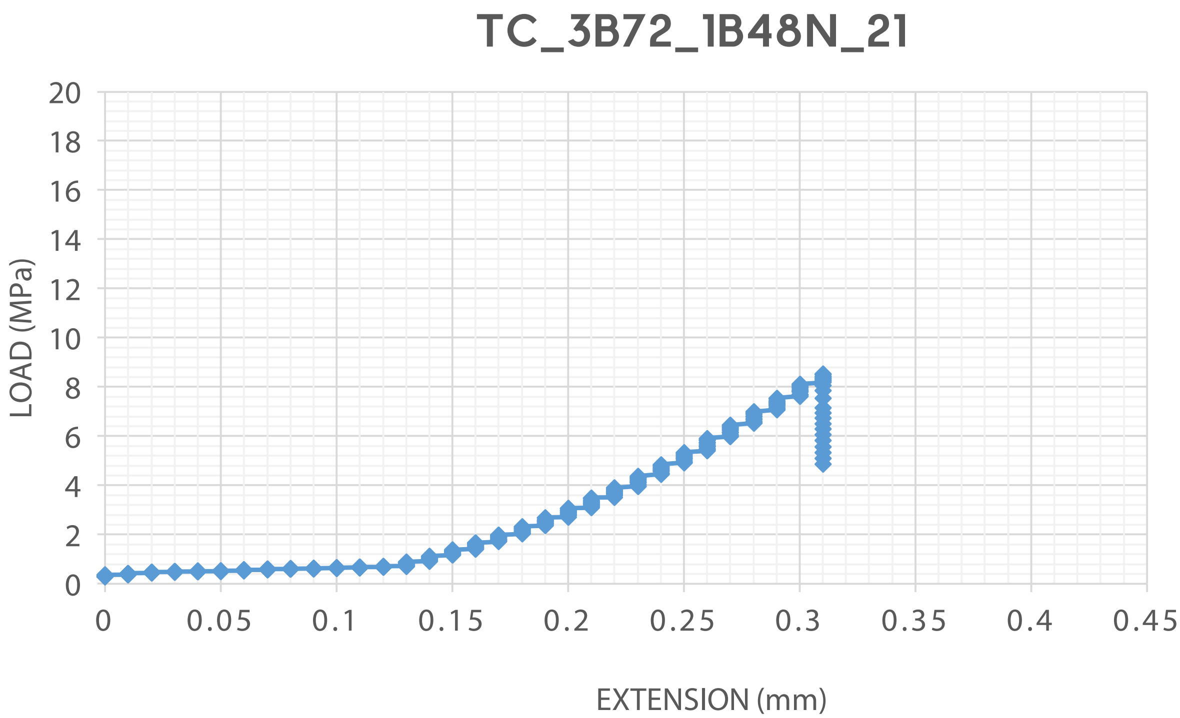
20
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_3B72_1B48N_21
LOAD (MPa)
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14
12
10
8
6
4
2
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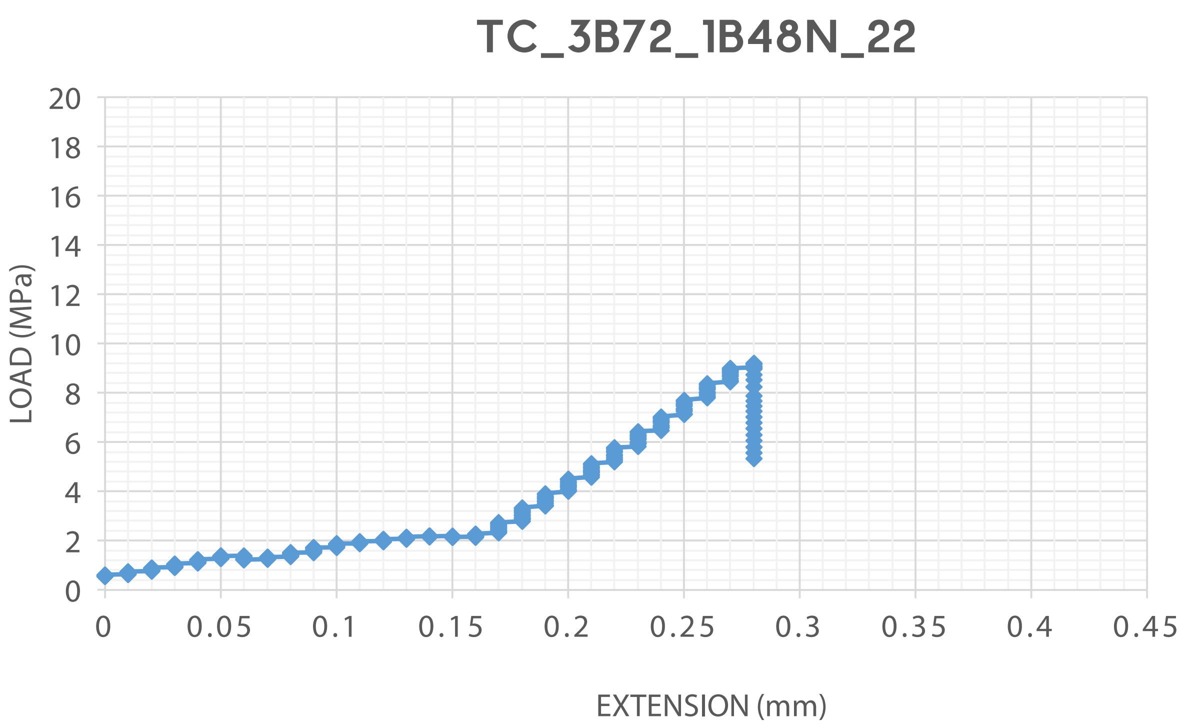
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_22
LOAD (MPa)
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12
10
8
6
4
2
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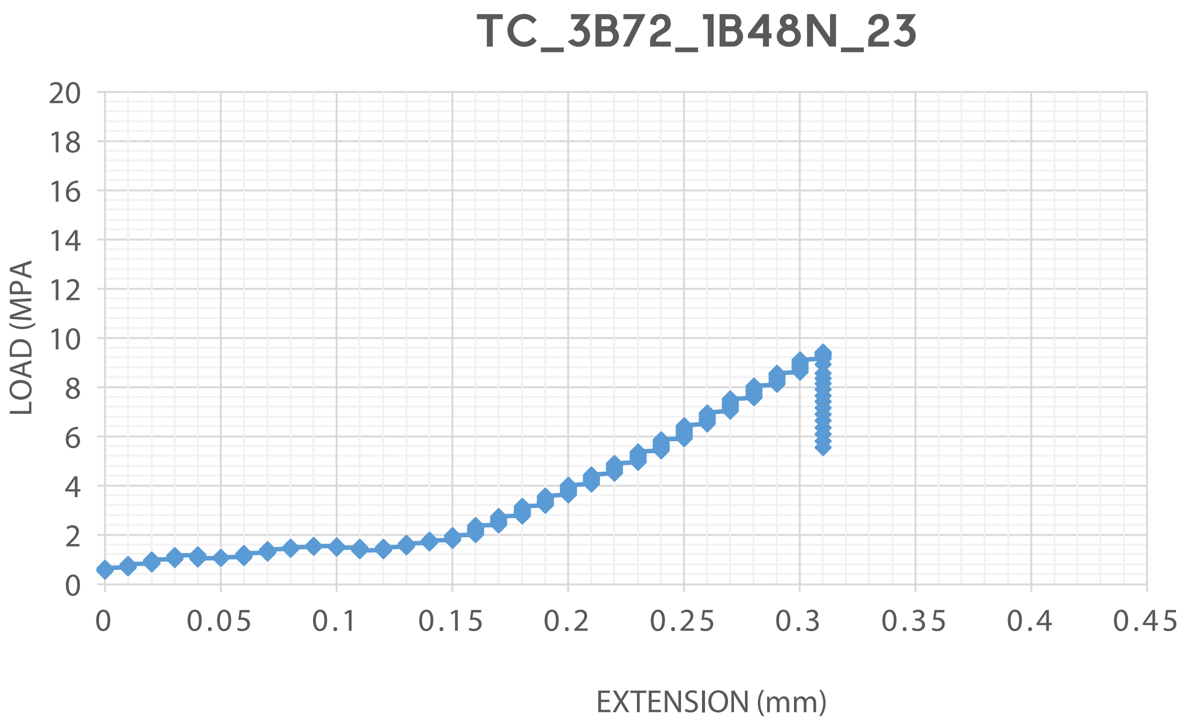
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_23
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6
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10
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16
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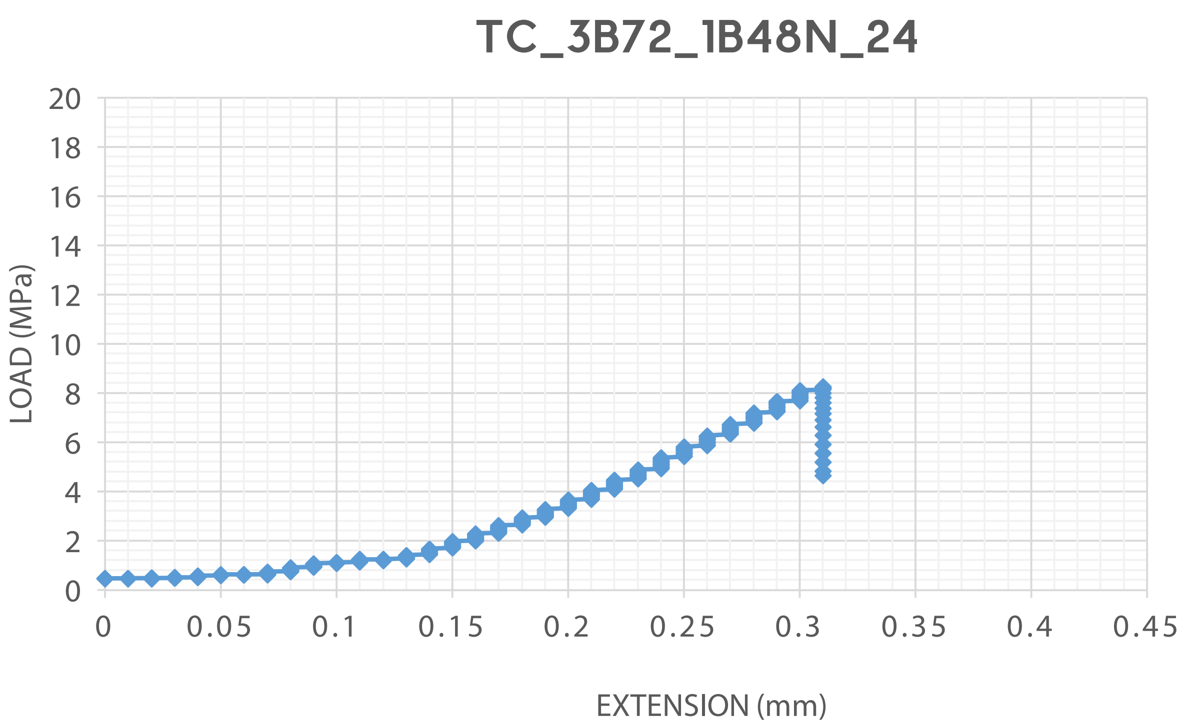
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_24
LOAD (MPa)
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16
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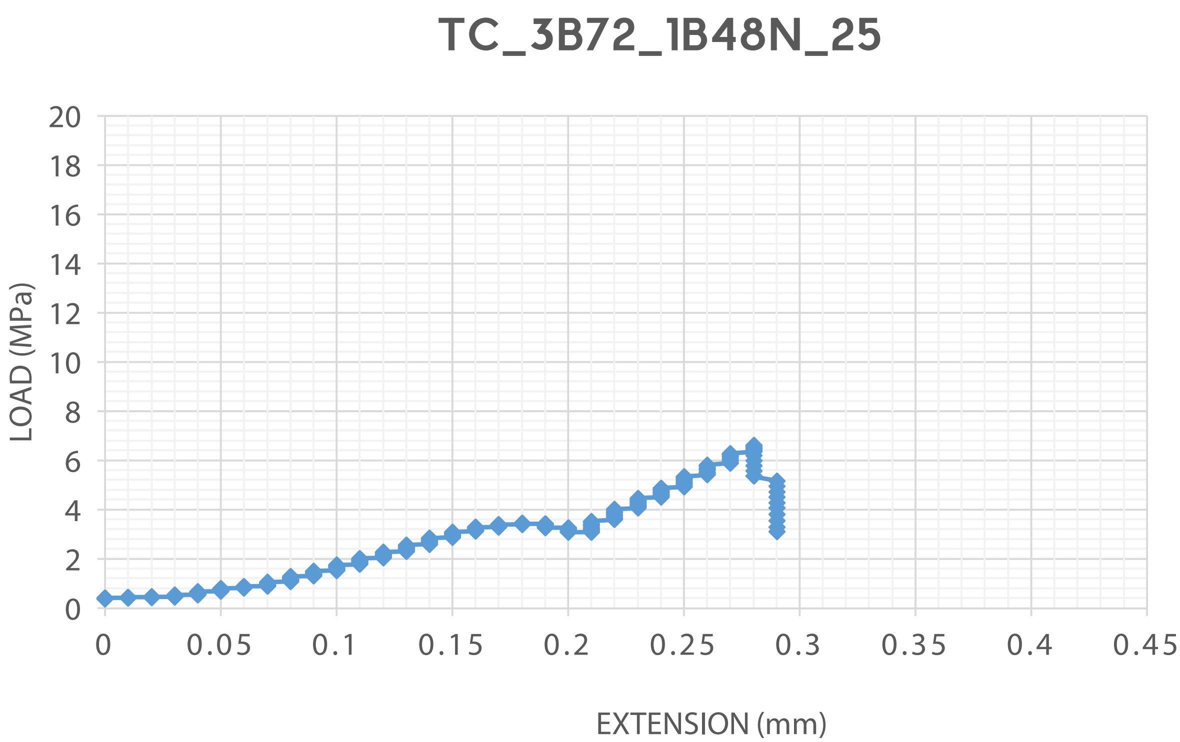
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)



TC_3B72_1B48N_25
LOAD (MPa)
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10
8
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4
2
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0
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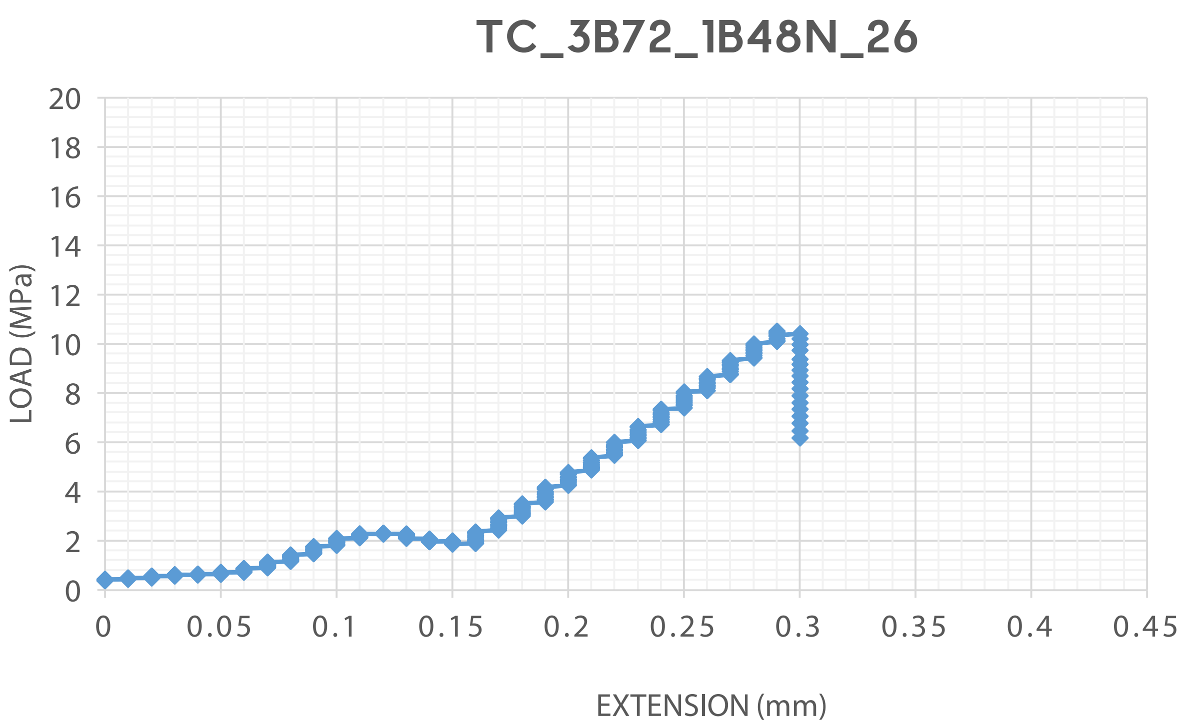
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)



TC_3B72_1B48N_26
LOAD (MPa)
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14
12
10
8
6
4
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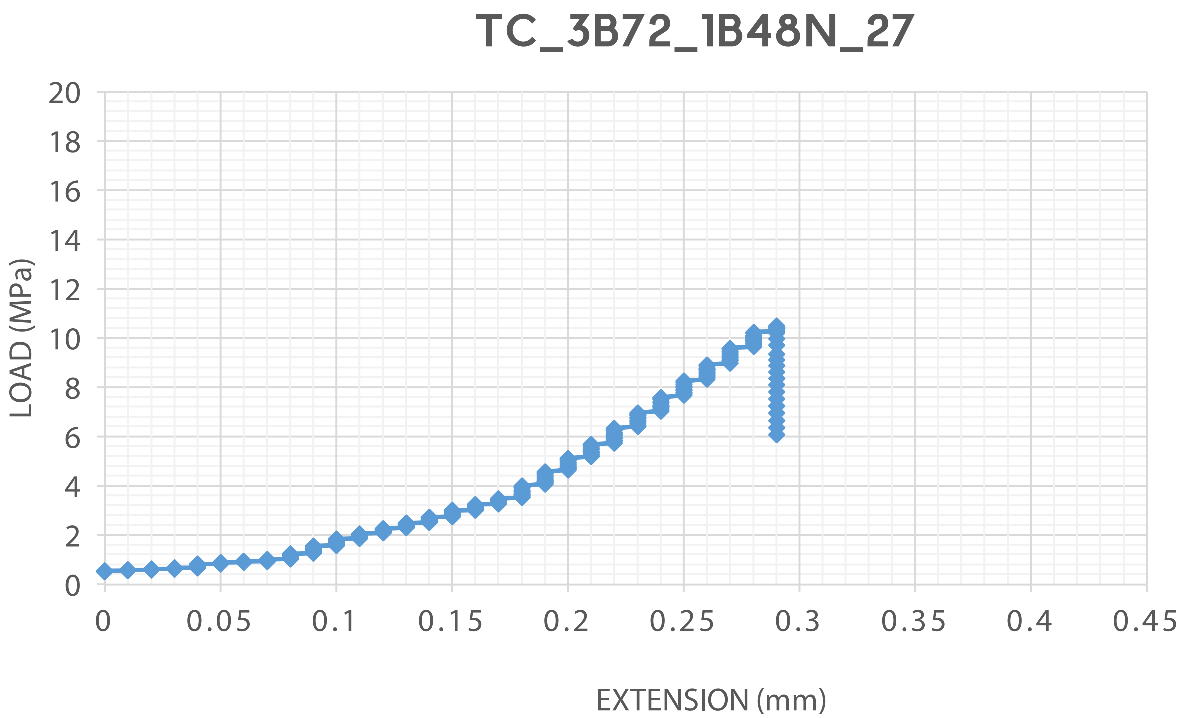
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_27
LOAD (MPa)
EXTENSION (mm)
0
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4
6
8
10
12
14
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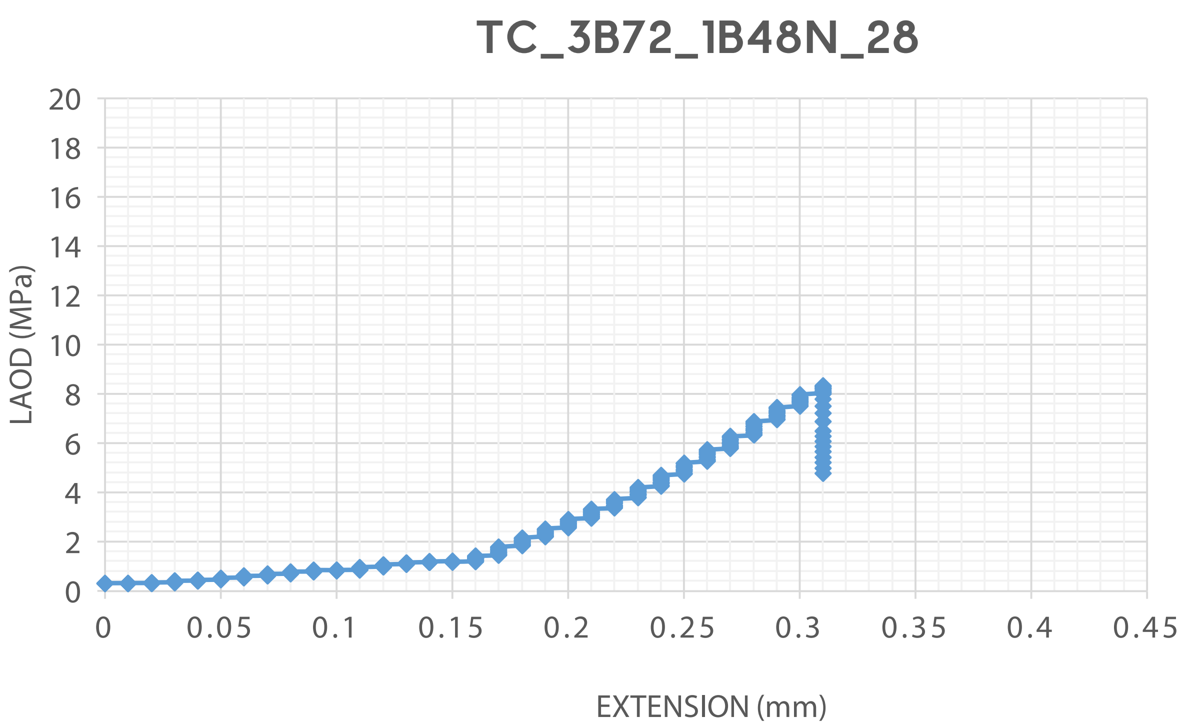
18
20
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_3B72_1B48N_28
LAOD (MPa)
EXTENSION (mm)
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4
6
8
10
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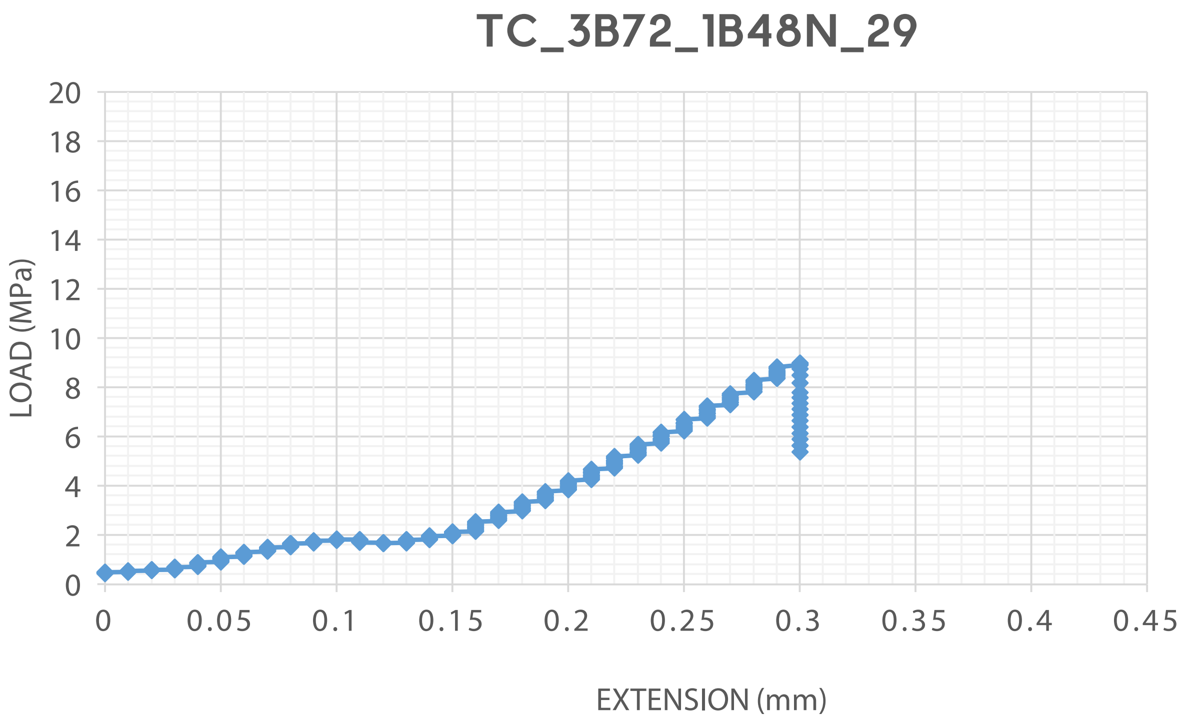
18
20
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_3B72_1B48N_29
LOAD (MPa)
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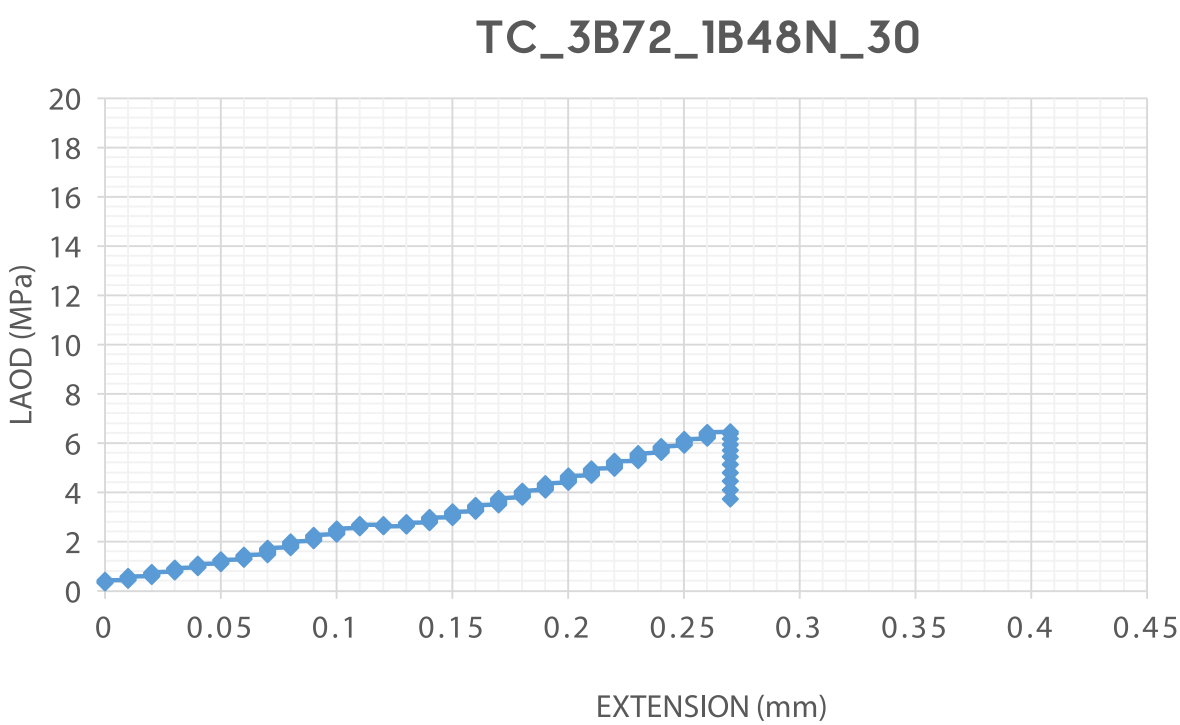
14
12
10
8
6
4
2
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_3B72_1B48N_30
LAOD (MPa)
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18
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14
12
10
8
6
4
2
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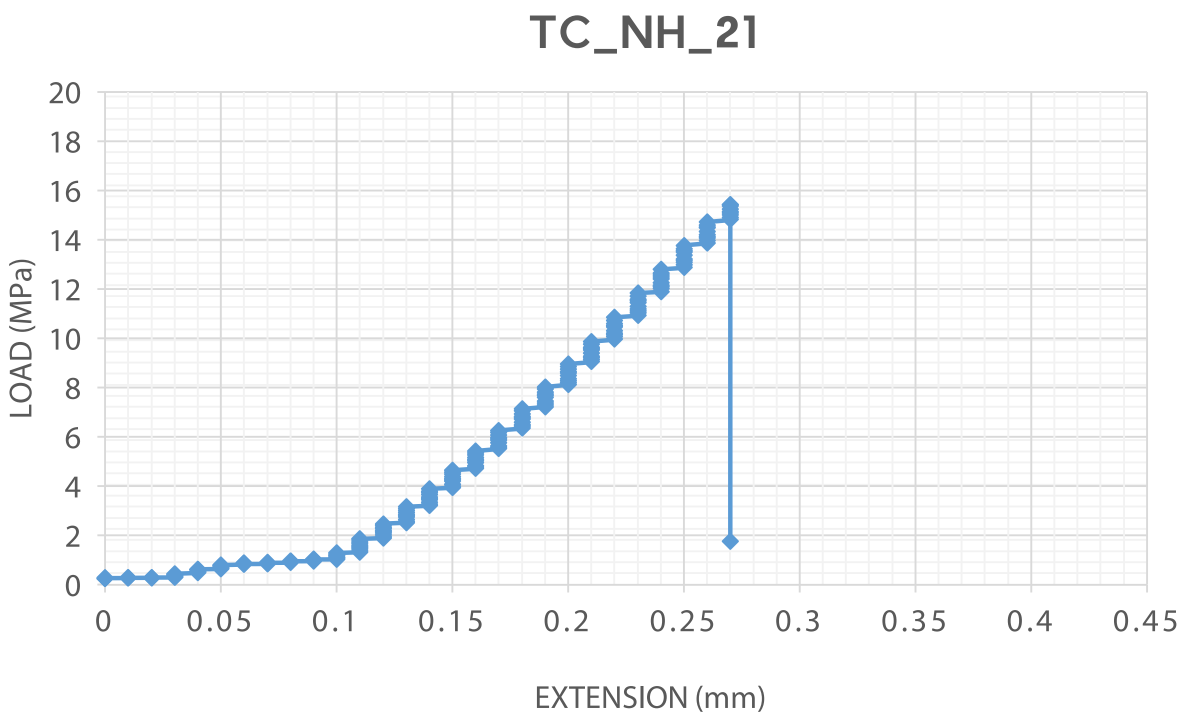
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NH_21
LOAD (MPa)
20
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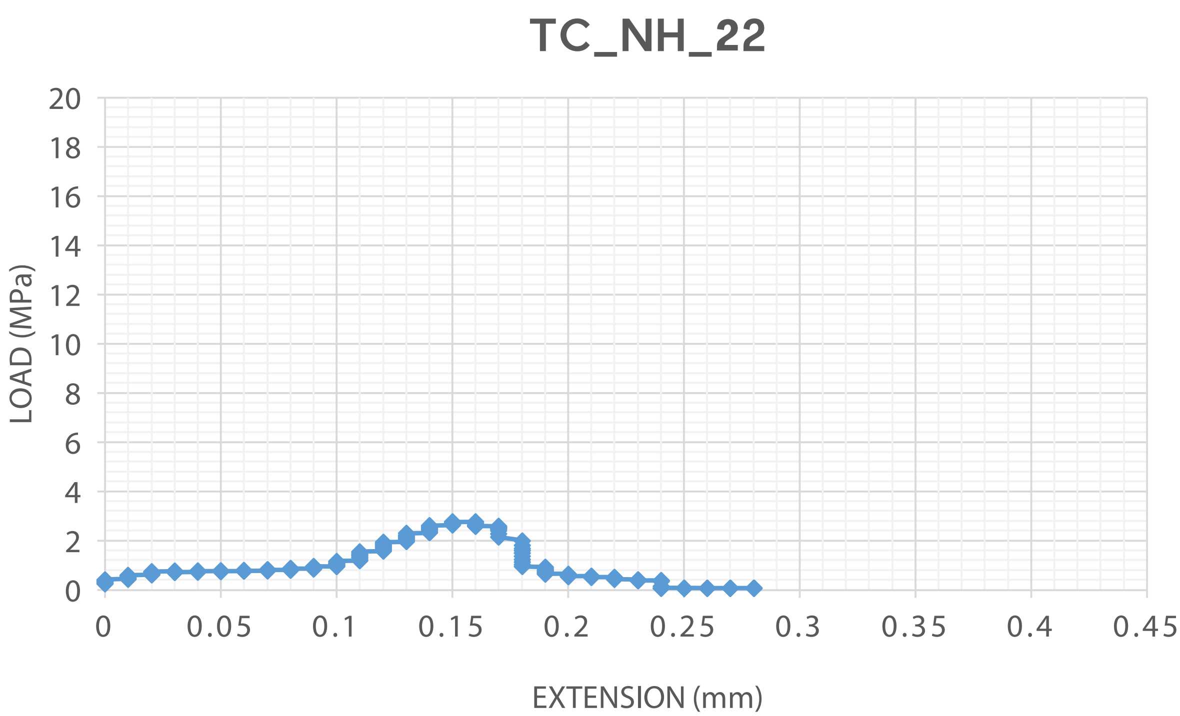
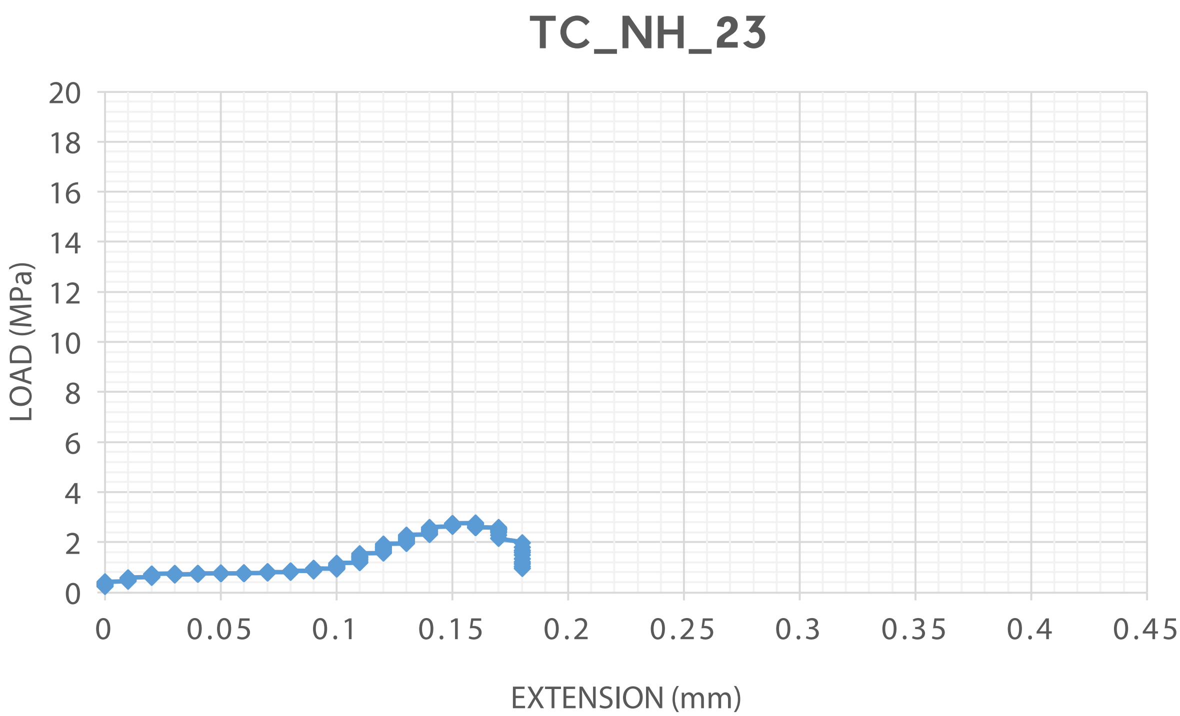
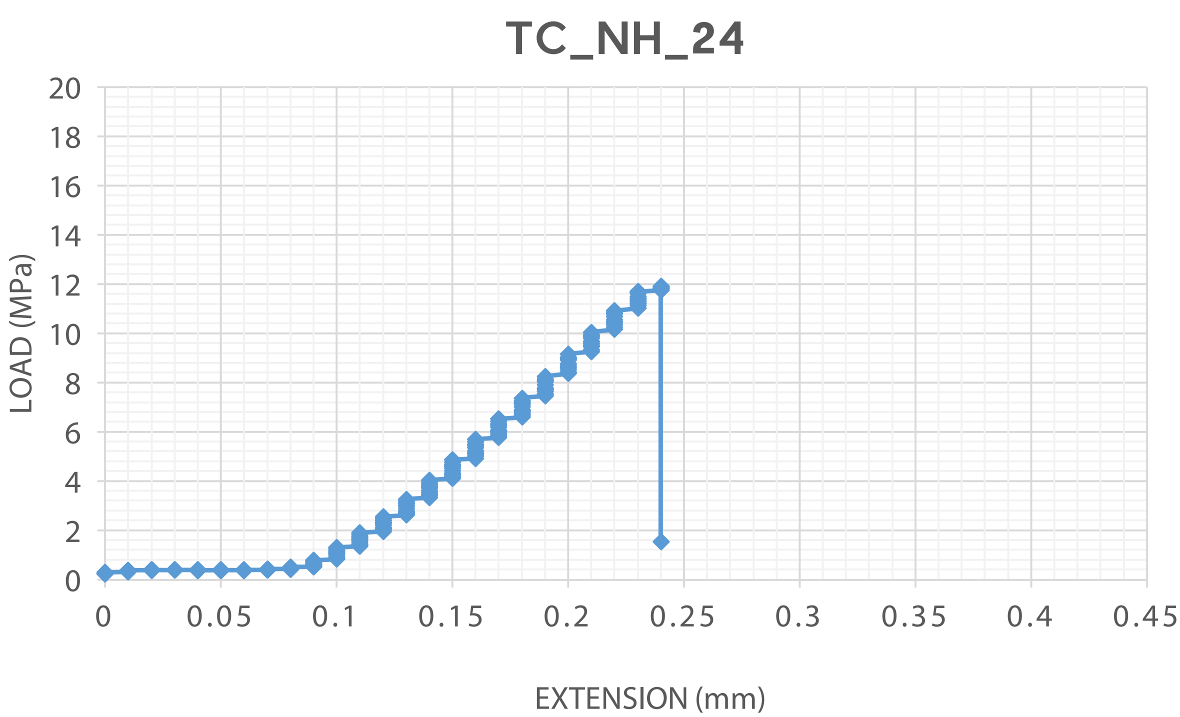
16
14
12
10
8
6
4
2
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NH_22
LOAD (MPa)
20
18
16
14
12
10
8
6
4
2
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)



TC_NH_23
20
18
16
14
12
10
8
6
4
2
0
LOAD (MPa)
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)



TC_NH_24
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18
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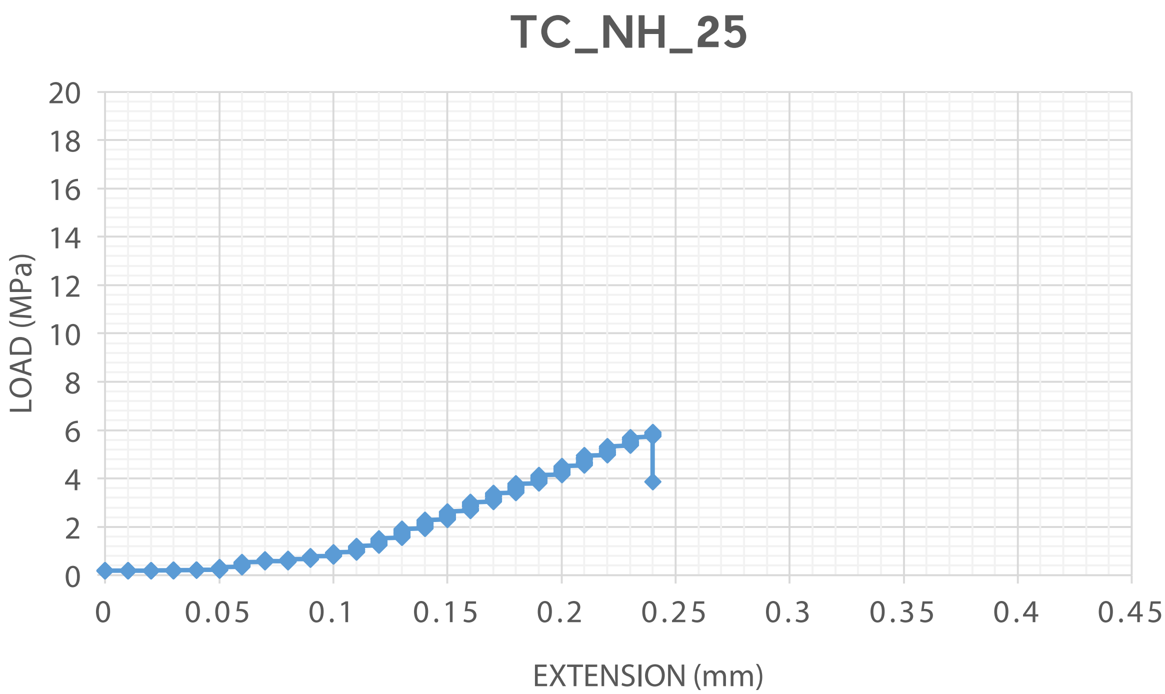
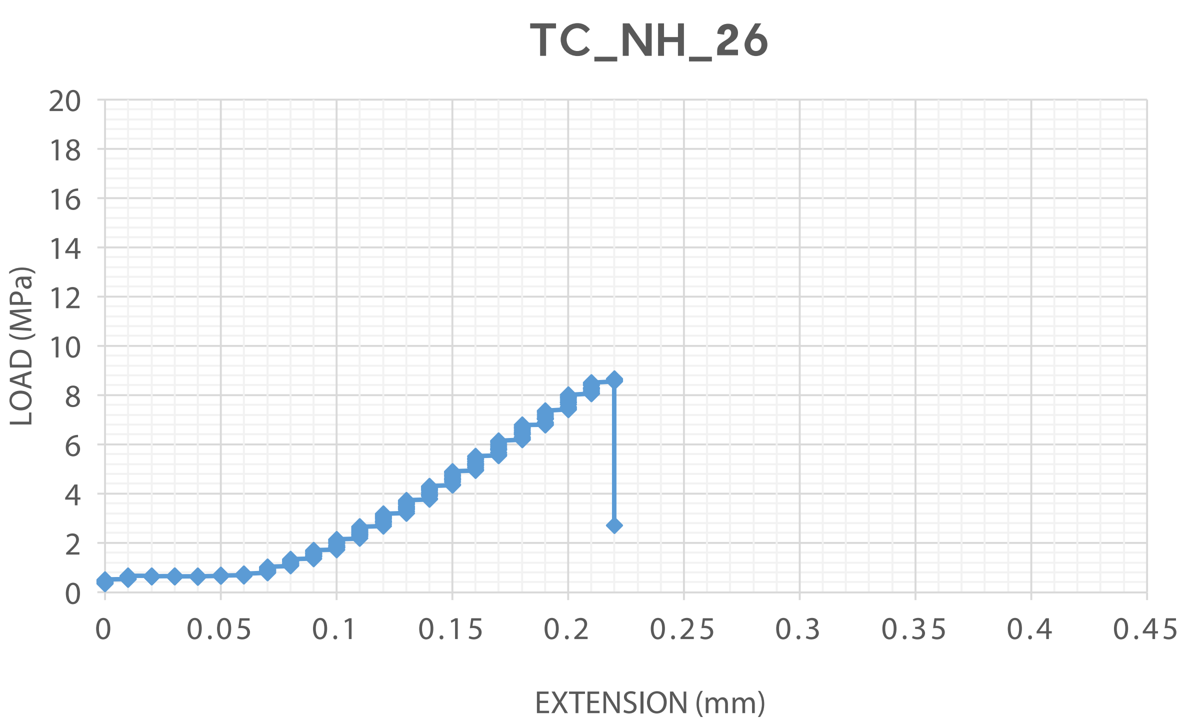
14
12
10
8
6
4
2
0
LOAD (MPa)
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NH_25
LOAD (MPa)
EXTENSION (mm)
0
2
4
6
8
10
12
14
16
18
20
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_NH_26
LOAD (MPa)
EXTENSION (mm)
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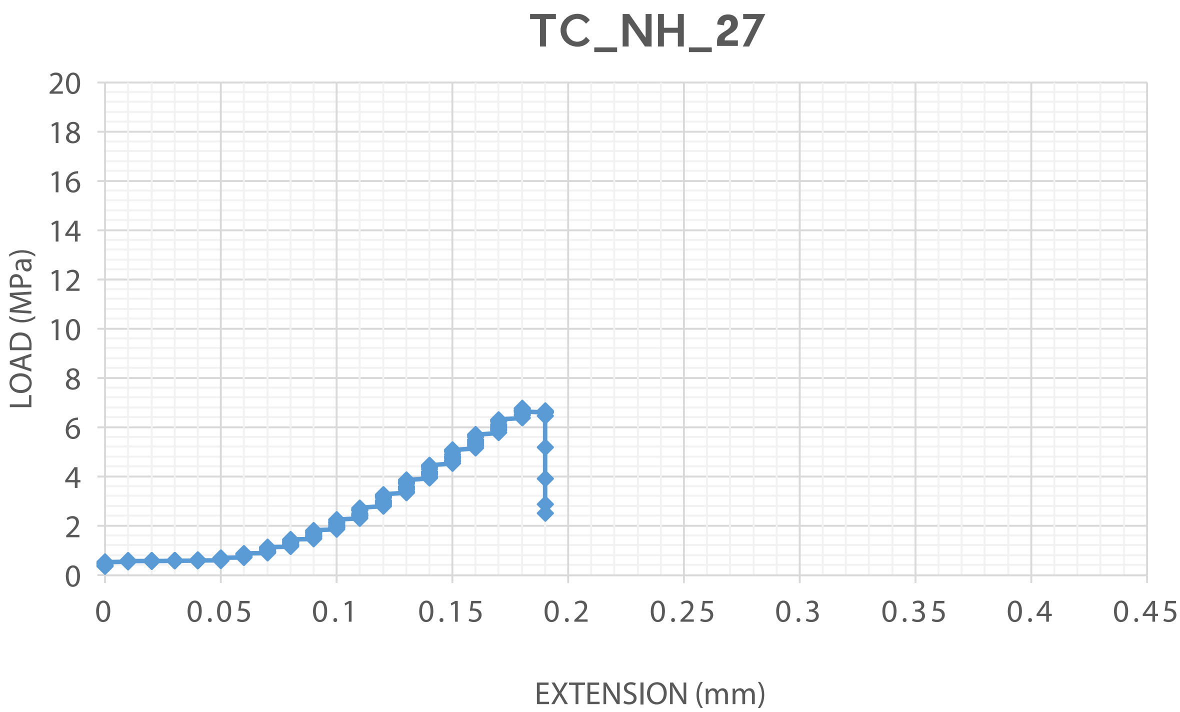
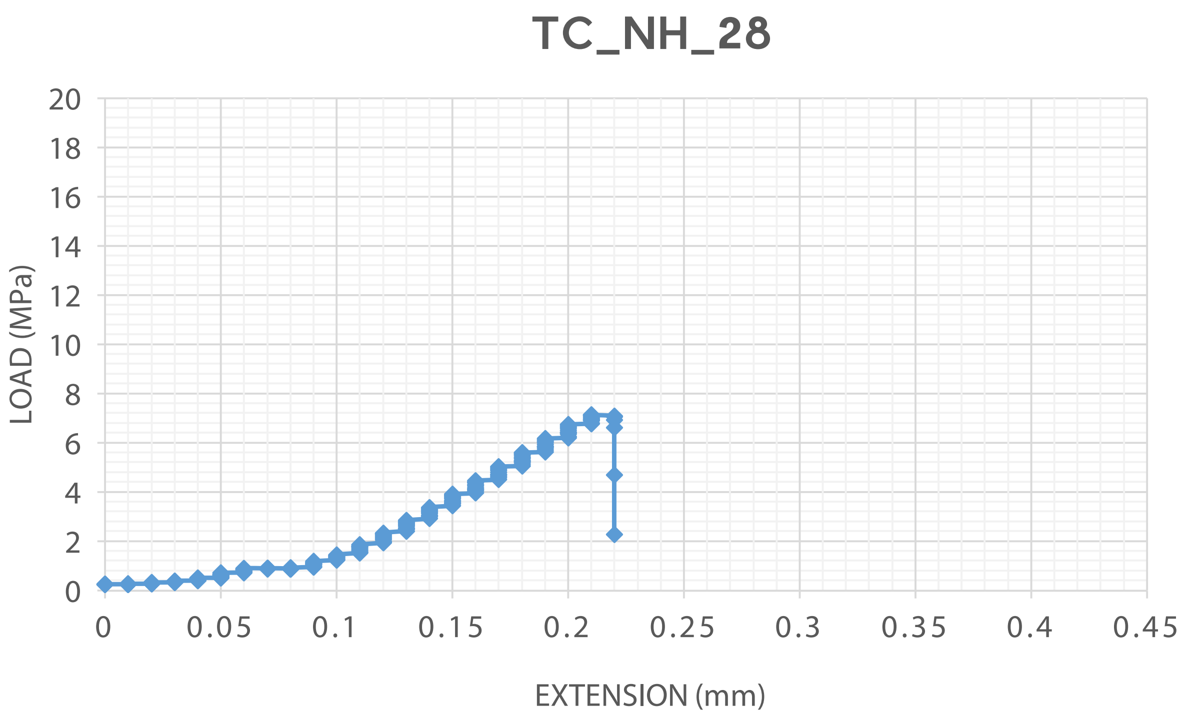
2
4
6
8
10
12
14
16
18
20
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45


TC_NH_27
LOAD (MPa)
20
18
16
14
12
10
8
6
4
2
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NH_28
LOAD (MPa)
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16
14
12
10
8
6
4
2
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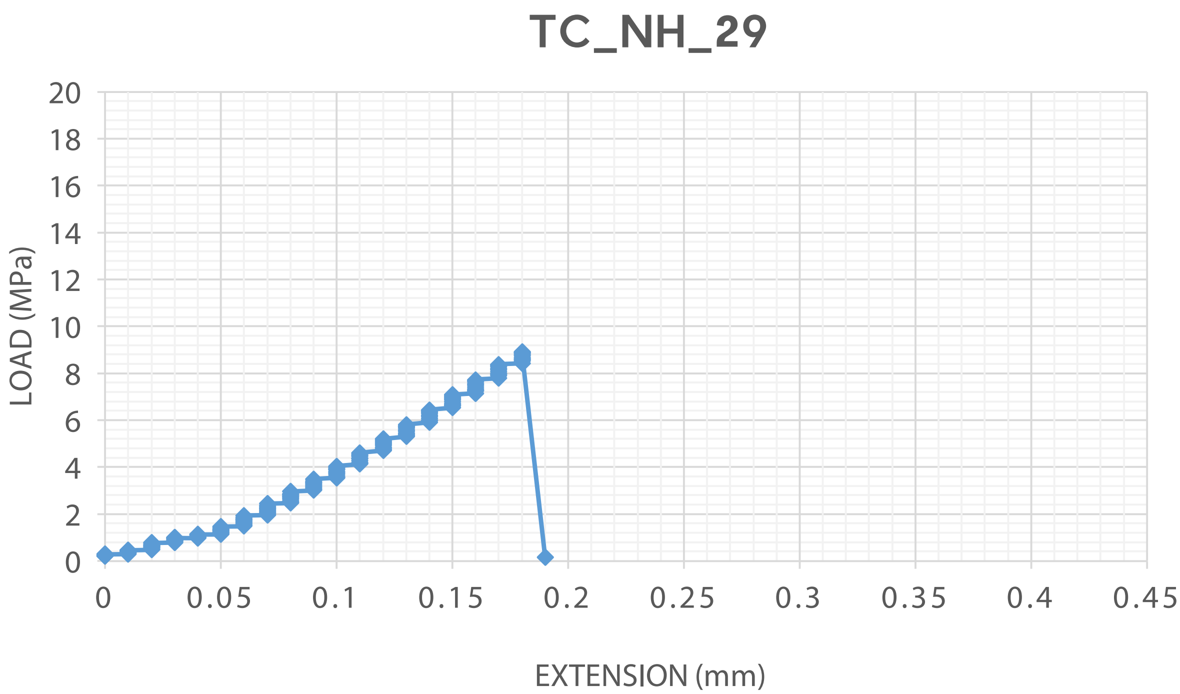
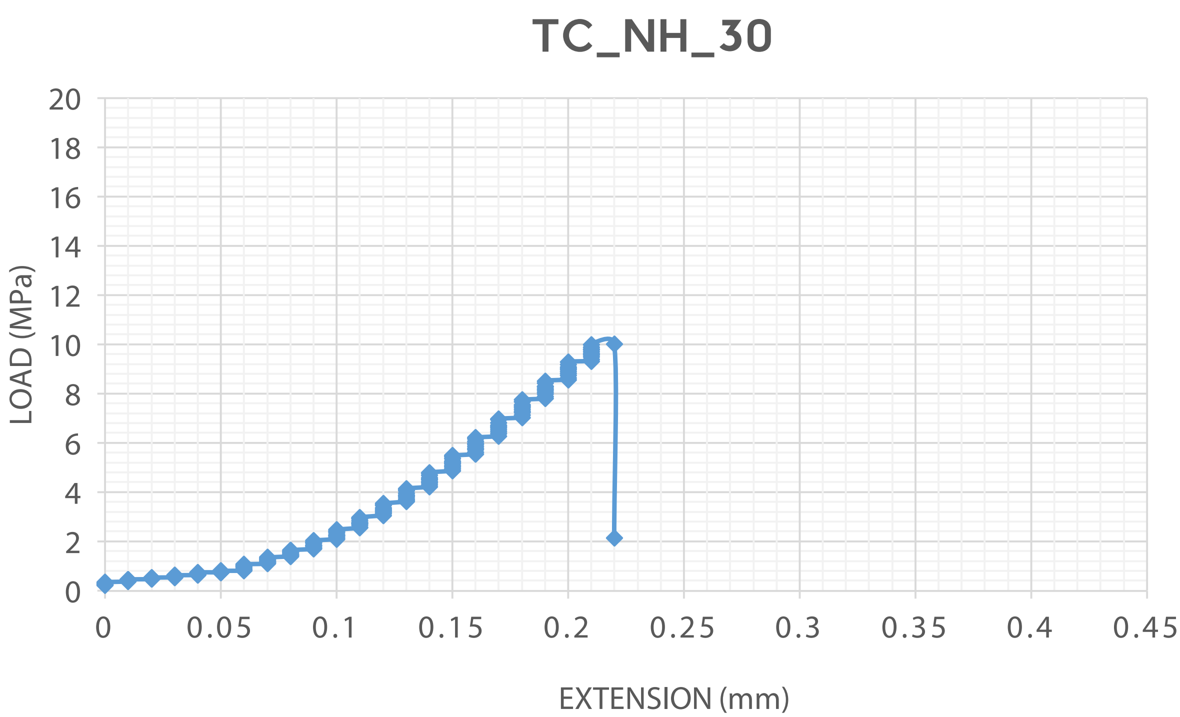
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NH_29
LOAD (MPa)
20
18
16
14
12
10
8
6
4
2
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)


TC_NH_30
LOAD (MPa)
20
18
16
14
12
10
8
6
4
2
0
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
EXTENSION (mm)

APPENDIX 4

LIMESTONE – GROUP B

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg. Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
L_B72_11	2/3/2017	3/13/2017	03/29/2017	4.26	32.6	50.5	Broke at joint - dripping and deflection				
L_B72_12	2/3/2017	3/13/2017	03/29/2017	4.47	30.5	51.8	Broke at joint				
L_B72_13	2/3/2017	3/13/2017	03/29/2017	3.18	34.6	49.6	Broke at joint				
L_B72_14	2/3/2017	3/13/2017	03/29/2017	3.07	32.1	48.0	did not break fully				
L_B72_15	2/3/2017	3/13/2017	03/29/2017	2.49	30.1	49.2	Broke at joint - dripping and deflection				
L_B72_16	2/3/2017	3/13/2017	03/29/2017	4.13	33.1	52.1	Broke at joint				
L_B72_17	2/3/2017	3/13/2017	03/29/2017	2.47	36.1	51.3	Broke at joint				
L_B72_18	2/3/2017	3/13/2017	03/29/2017	3.58	38.5	55.3	Broke at joint				
L_B72_19	2/3/2017	3/13/2017	03/29/2017	3.22	33.8	50.8	Broke at joint				
L_B72_20	2/3/2017	3/13/2017	03/29/2017	4.16	33.6	51.8	Broke at joint				
								3.50	51.04	0.731	1.987
L_B48N_11	2/3/2017	3/13/2017	03/30/2017	5.21	40.9	63.5	Broke at joint				
L_B48N_12	2/3/2017	3/13/2017	03/30/2017	5.20	42.2	64.8	Broke at joint				
L_B48N_13	2/3/2017	3/13/2017	03/30/2017	6.20	39.9	64.7	Broke at joint				
L_B48N_14	2/3/2017	3/13/2017	03/30/2017	5.34	44.8	64.6	Broke at joint				
L_B48N_15	2/3/2017	3/13/2017	03/30/2017	5.20	44.2	64.4	Broke at joint				
L_B48N_16	2/3/2017	3/13/2017	03/30/2017	3.03	28.5	48.9	Broke at joint				

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg. Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
L_B48N_17	2/3/2017	3/13/2017	03/30/2017	5.57	33.3	56.3	Broke at joint - dripping and deflection				
L_B48N_18	2/3/2017	3/13/2017	03/30/2017	5.38	38.5	59.7	didn't break completely				
L_B48N_19	2/3/2017	3/13/2017	03/30/2017	6.46	42.1	63.2	Broke at joint				
L_B48N_20	2/3/2017	3/13/2017	03/30/2017	5.55	45.4	63.4	Broke at joint				
								5.31	61.35	0.910	5.140
L_B44_11	2/3/2017	3/13/2017	03/30/2017	8.45	40.0	67.7	Broke at joint				
L_B44_12	2/3/2017	3/13/2017	03/30/2017	5.41	45.7	62.9	Broke at joint				
L_B44_13	2/3/2017	3/13/2017	03/30/2017	7.05	47.0	67.9	Broke at joint				
L_B44_14	2/3/2017	3/13/2017	03/30/2017	7.12	48.8	63.5	Broke at joint				
L_B44_15	2/3/2017	3/13/2017	03/30/2017	6.08	43.1	64.8	Broke at joint				
L_B44_16	2/3/2017	3/13/2017	03/30/2017	7.40	43.1	65.2	Broke at joint				
L_B44_17	2/3/2017	3/13/2017	03/30/2017	6.13	55.0	67.8	Broke at joint				
L_B44_18	2/3/2017	3/13/2017	03/30/2017	6.31	49.0	66.3	Broke at joint				
L_B44_19	2/3/2017	3/13/2017	03/30/2017	7.41	34.6	63	Broke at joint - dripping and deflection				
L_B44_20	2/3/2017	3/13/2017	03/30/2017	8.03	46.3	65.1					
								6.939	65.42	0.947	1.950
L_A11_11	2/3/2017	3/13/2017	03/30/2017	13.35	46.1	76.6	Broke at joint				
L_A11_12	2/3/2017	3/13/2017	03/30/2017	19.29	47.9	75.3	Broke at joint				
L_A11_13	2/3/2017	3/13/2017	03/30/2017	21.10	54.6	80.5	Broke at joint				

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg. Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
L_A11_14	2/3/2017	3/13/2017	03/30/2017	21.29	54.6	79.6	Broke at joint				
L_A11_15	2/3/2017	3/13/2017	03/30/2017	14.25	51.3	76.4	Broke at joint				
L_A11_16	2/3/2017	3/13/2017	04/03/2017	18.23	47.9	76.8	Broke at joint				
L_A11_17	2/3/2017	3/13/2017	04/03/2017	N/A	N/A	N/A	broke immediately				
L_A11_18	2/3/2017	3/13/2017	04/03/2017	14.19	50.1	77.9	Broke at joint				
L_A11_19	2/3/2017	3/13/2017	04/03/2017	14.02	51.2	76.9	Broke at joint				
L_A11_20	2/3/2017	3/13/2017	04/03/2017	25.00	52.9	N/A	did not break fully				
								17.857	77.5	4.138	1.744
L_1B72_3B48N_11	2/3/2017	3/13/2017	03/30/2017	7.48	27.4	56.5	Broke at joint				
L_1B72_3B48N_12	2/3/2017	3/13/2017	03/30/2017	4.41	40.1	58.0	Broke at joint				
L_1B72_3B48N_13	2/3/2017	3/13/2017	03/30/2017	5.44	40.9	62.6	Broke at joint				
L_1B72_3B48N_14	2/3/2017	3/13/2017	03/30/2017	5.50	41.8	61.9	Broke at joint				
L_1B72_3B48N_15	2/3/2017	3/13/2017	03/30/2017	6.01	44.3	63.8	Broke at joint				
L_1B72_3B48N_16	2/3/2017	3/13/2017	03/30/2017	5.36	46.3	63.8	Broke at joint				
L_1B72_3B48N_17	2/3/2017	3/13/2017	03/30/2017	6.03	41.8	64.4	Broke at joint				
L_1B72_3B48N_18	2/3/2017	3/13/2017	03/30/2017	6.35	38.5	63.2	Broke at joint				
L_1B72_3B48N_19	2/3/2017	3/13/2017	03/30/2017	5.28	46.0	64.2	Broke at joint				
L_1B72_3B48N_20	2/3/2017	3/13/2017	03/30/2017	6.11	47.1	66.3	Broke at joint				
								5.797	62.47	0.812	3.008
L_3B72_1B48N_11	2/3/2017	3/13/2017	03/29/2017	5.20	38.9	63.1	Broke at joint				
L_3B72_1B48N_12	2/3/2017	3/13/2017	03/29/2017	3.58	41.6	61.1	Broke at joint				
L_3B72_1B48N_13	2/3/2017	3/13/2017	03/29/2017	4.43	44.6	62.6	Broke at joint				

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg. Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
L_3B72_1B48N_14	2/3/2017	3/13/2017	03/29/2017	4.40	47.5	64.4	did not break fully				
L_3B72_1B48N_15	2/3/2017	3/13/2017	03/29/2017	3.22	35.3	61.2	Broke at joint				
L_3B72_1B48N_16	2/3/2017	3/13/2017	03/29/2017	5.13	43.5	64.0	Broke at joint				
L_3B72_1B48N_17	2/3/2017	3/13/2017	03/29/2017	4.21	38.3	63.7	Broke at joint				
L_3B72_1B48N_18	2/3/2017	3/13/2017	03/29/2017	3.34	42.2	62.1	Broke at joint				
L_3B72_1B48N_19	2/3/2017	3/13/2017	03/29/2017	3.53	45.3	61.4	Broke at joint				
L_3B72_1B48N_20	2/3/2017	3/13/2017	03/29/2017	4.35	47.1	63.8	Broke at joint				
								4.139	62.74	0.705	1.238
L_CT_NA_11	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_12	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_13	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_14	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_15	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_16	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_17	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_18	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_19	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
L_CT_NA_20	N/A	N/A	03/29/2017	N/A	N/A	N/A	Did not break				
								N/A	N/A	N/A	N/A

TERRA COTTA – GROUP B

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg .Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
TC_B72_11	2/1/2017	3/13/2017	03/30/2017	4.47	42.7	60.9	Broke at joint				
TC_B72_12	2/1/2017	3/13/2017	03/30/2017	4.56	45.8	62.1	Broke at joint				
TC_B72_13	2/1/2017	3/13/2017	03/30/2017	4.55	45.2	62.5	Broke at joint				
TC_B72_14	2/1/2017	3/13/2017	03/30/2017	5.58	47.4	67.8	Broke at joint				
TC_B72_15	2/1/2017	3/13/2017	03/30/2017	5.27	47.6	64	Broke at joint				
TC_B72_16	2/1/2017	3/13/2017	03/30/2017	5.37	50.2	64.9	Broke at joint				
TC_B72_17	2/1/2017	3/13/2017	03/30/2017	3.35	45.2	61.0	Broke at joint				
TC_B72_18	2/1/2017	3/13/2017	03/30/2017	3.47	47.0	61.7	Broke at joint				
TC_B72_19	2/1/2017	3/13/2017	03/30/2017	5.58	49.0	64.3	Broke at joint				
TC_B72_20	2/1/2017	3/13/2017	03/30/2017	4.38	46.2	61.9	Broke at joint				
								4.65	63.11	0.80	2.14
TC_B48N_11	2/2/2017	3/13/2017	04/03/2017	7.03	47.7	68.0	Broke at joint				
TC_B48N_12	2/2/2017	3/13/2017	04/03/2017	5.19	47.3	65.8	Broke at joint				
TC_B48N_13	2/2/2017	3/13/2017	04/03/2017	7.16	48.5	68.8	Broke at joint				
TC_B48N_14	2/2/2017	3/13/2017	04/03/2017	6.02	52.6	64.7	Broke at joint				
TC_B48N_15	2/2/2017	3/13/2017	04/03/2017	5.00	51.9	66.3	Broke at joint				
TC_B48N_16	2/2/2017	3/13/2017	04/03/2017	6.53	47.1	67.7	Broke at joint				
TC_B48N_17	2/2/2017	3/13/2017	04/03/2017	6.48	48.0	68.1	Broke at joint				
TC_B48N_18	2/2/2017	3/13/2017	04/03/2017	6.07	50.3	68.0	Broke at joint				
TC_B48N_19	2/2/2017	3/13/2017	04/03/2017	9.51	48.0	70.8	Broke at joint				
TC_B48N_20	2/2/2017	3/13/2017	04/03/2017	6.18	46.6	68.4	Broke at joint				

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg .Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
								6.51	67.66	1.25	1.70
TC_B44_11	2/2/2017	3/13/2017	03/30/2017	7.08	48.6	67.6	Broke at joint				
TC_B44_12	2/2/2017	3/13/2017	03/30/2017	7.05	43.0	64.9	Broke at joint				
TC_B44_13	2/2/2017	3/13/2017	03/30/2017	6.29	51.1	65.5	Broke at joint				
TC_B44_14	2/2/2017	3/13/2017	03/30/2017	6.16	51.2	67.3	Broke at joint				
TC_B44_15	2/2/2017	3/13/2017	03/30/2017	6.27	51.8	65.0	Broke at joint				
TC_B44_16	2/2/2017	3/13/2017	03/30/2017	7.54	51.0	66.3	Broke at joint				
TC_B44_17	2/2/2017	3/13/2017	03/30/2017	5.36	51.1	64.9	Broke at joint				
TC_B44_18	2/2/2017	3/13/2017	03/30/2017	7.48	48.1	67.1	Broke at joint				
TC_B44_19	2/2/2017	3/13/2017	03/30/2017	6.22	51.1	66.5	Broke at joint				
TC_B44_20	2/2/2017	3/13/2017	03/30/2017	5.27	53.0	65.3	Broke at joint				
								6.47	66.04	0.80	1.05
TC_A11_11	2/2/2017	3/13/2017	04/03/2017	25.00	51.6	N/A	20 lb				
TC_A11_12	2/2/2017	3/13/2017	04/03/2017	11.3	56.5	73.8	25 lb				
TC_A11_13	2/2/2017	3/13/2017	04/03/2017	7.03	54.8	71.6	25 lb				
TC_A11_14	2/2/2017	3/13/2017	04/03/2017	17.56	54.1	76.0	25 lb				
TC_A11_15	2/2/2017	3/13/2017	04/03/2017	5.53	68.0	75.4	25 lb				
TC_A11_16	2/2/2017	3/13/2017	04/03/2017	17.47	51.8	79.6	25 lb - did not break fully				
TC_A11_17	2/2/2017	3/13/2017	04/03/2017	14.4	51.8	74.0	25 lb - did not break fully				
TC_A11_18	2/2/2017	3/13/2017	04/04/2017	13.10	29.5	70.3	25 lb				
TC_A11_19	2/2/2017	3/13/2017	04/04/2017	23.03	45.2	77.1	25 lb				

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg .Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
TC_A11_20	2/2/2017	3/13/2017	04/04/2017	10.29	52.0	71.7	25 lb				
								14.47	74.38	6.38	2.96
TC_1B72_3B48N_11	2/2/2017	3/13/2017	03/30/2017	5.56	49.1	66.6	Broke at joint				
TC_1B72_3B48N_12	2/2/2017	3/13/2017	03/30/2017	6.03	47.1	67.1	Broke at joint				
TC_1B72_3B48N_13	2/2/2017	3/13/2017	03/30/2017	5.26	48.4	65.9	Broke at joint				
TC_1B72_3B48N_14	2/2/2017	3/13/2017	04/03/2017	6.47	29.8	57.5	Broke at joint				
TC_1B72_3B48N_15	2/2/2017	3/13/2017	04/03/2017	5.47	41.8	61.6	Broke at joint				
TC_1B72_3B48N_16	2/2/2017	3/13/2017	04/03/2017	6.33	45.2	63.1	Broke at joint				
TC_1B72_3B48N_17	2/2/2017	3/13/2017	04/03/2017	5.36	44.4	64.1	Broke at joint				
TC_1B72_3B48N_18	2/2/2017	3/13/2017	04/03/2017	5.51	47.6	64.7	Broke at joint				
TC_1B72_3B48N_19	2/2/2017	3/13/2017	04/03/2017	6.25	46.1	64.1	Broke at joint				
TC_1B72_3B48N_20	2/2/2017	3/13/2017	04/03/2017	5.40	46.6	65.7	Broke at joint				
								5.76	64.04	0.45	2.83
TC_3B72_1B48N_11	2/2/2017	3/13/2017	03/30/2017	4.41	47.7	64.1	Broke at joint				
TC_3B72_1B48N_12	2/2/2017	3/13/2017	03/30/2017	4.52	48.1	64.5	Broke at joint				
TC_3B72_1B48N_13	2/2/2017	3/13/2017	03/30/2017	6.13	48.3	64.5	Broke at joint				
TC_3B72_1B48N_14	2/2/2017	3/13/2017	03/30/2017	4.48	49.1	64.3	Broke at joint				
TC_3B72_1B48N_15	2/2/2017	3/13/2017	03/30/2017	3.36	50.1	62.1	Broke at joint				
TC_3B72_1B48N_16	2/2/2017	3/13/2017	03/30/2017	5.23	49.4	64.3	Broke at joint				
TC_3B72_1B48N_17	2/2/2017	3/13/2017	03/30/2017	4.11	46.5	65.1	Broke at joint				
TC_3B72_1B48N_18	2/2/2017	3/13/2017	03/30/2017	6.25	47.6	65.7	Broke at joint				
TC_3B72_1B48N_19	2/2/2017	3/13/2017	03/30/2017	3.50	50.4	62.3	Broke at joint				

	Bonding Start Date	Bonding End Date	Testing Date	Time till Failure	Beginning Temp. °C	Temp. at Failure °C	Notes	Avg. Time at Failure	Avg .Temp at Failure	Standard Deviation for Time	Standard Deviation for Temp
TC_3B72_1B48N_20	2/2/2017	3/13/2017	03/30/2017	4.52	50.6	62.9	Broke at joint				
								4.65	63.98	0.97	1.17
TC_CT_NA_11	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_12	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_13	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_14	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_15	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_16	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_17	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_18	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_19	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
TC_CT_NA_20	N/A	N/A	03/30/2017	N/A	N/A	N/A	Did not break				
								N/A	N/A	N/A	N/A